

Research Paper

Dielectric Properties in the $\text{Pb}_{1-3x/2}\text{La}_x[(\text{Mg}_{1/3}\text{Ta}_{2/3})_{0.66}\text{Zr}_{0.34}]\text{O}_3$ Systems

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Abstract The dielectric constant and loss of poling/non-poling was measured in the $\text{Pb}_{1-3x/2}\text{La}_x[(\text{Mg}_{1/3}\text{Ta}_{2/3})_{0.66}\text{Zr}_{0.34}]\text{O}_3$ samples. The addition of La^{3+} to the $\text{Pb}_{1-3x/2}\text{La}_x[(\text{Mg}_{1/3}\text{Ta}_{2/3})_{0.66}\text{Zr}_{0.34}]\text{O}_3$ did not cause a large change in grain size. But the addition of La^{3+} did show transition temperature, which shifted toward low temperature in the $\text{Pb}[(\text{Mg}_{1/3}\text{Ta}_{2/3})\text{Zr}]\text{O}_3$ systems. In addition, the dielectric and pyroelectric properties ($\epsilon \sim 20000$, $p \sim 0.03 \text{ C/m}^2\text{K}$) of this system using La^{3+} have been greatly improved. Pyroelectrics $\text{Pb}_{0.97}\text{La}_{0.02}[(\text{Mg}_{1/3}\text{Ta}_{2/3})_{0.66}\text{Zr}_{0.34}]\text{O}_3$ system was found to have a relatively high ferroelectric FOMs ($F_V \sim 0.035 \text{ m}^2/\text{C}$, $F_D \sim 0.52 \times 10^{-4} \text{ Pa}^{-1/2}$) at room temperature. Spontaneous polarization showed a value of $0.27 \sim 0.35 \text{ C/m}^2$ in the composition added to La^{3+} . The piezoelectric constant ($d_{33} = 350 \sim 490 \text{ pC/N}$) and electromechanical coupling factor ($k_p = 0.25 \sim 0.35$) are obtained in $\text{Pb}_{1-3x/2}\text{La}_x[(\text{Mg}_{1/3}\text{Ta}_{2/3})_{0.66}\text{Zr}_{0.34}]\text{O}_3$ compositions with La^{3+} dopant.

Keywords: $\text{Pb}_{1-3x/2}\text{La}_x[(\text{Mg}_{1/3}\text{Ta}_{2/3})_{0.66}\text{Zr}_{0.34}]\text{O}_3$, Ferroelectric, Relaxor, Pyroelectric

I. Introduction

Relaxor ferroelectrics crystallize to the general formula $\text{Pb}(\text{B}_1\text{B}_2)\text{O}_3$, where B_1 is a low valence cation and B_2 is a high valence cation, can undergo at least short-range ordering of (B_1B_2) -site cations. Lead magnesium tantalate $\text{Pb}(\text{Mg}_{1/3}\text{Ta}_{2/3})\text{O}_3$, a relaxor ferroelectric with a broad anomaly in dielectric constant centered around a maximum at -98°C [1]. The anti-ferroelectricity of lead zirconate PbZrO_3 with a Curie temperature of 230°C is indicated by a "missed" incommensurate state [2]. Anti-ferroelectric is a material in which neighbor spontaneous polarization dipoles are anti-parallel to each other. When $\text{Pb}(\text{Mg}_{1/3}\text{Ta}_{2/3})\text{O}_3$ and PbZrO_3 form a solid solution, the phase transition temperature and electrical characteristics depend on the composition. It was previously reported that solid solutions of $\text{Pb}[(\text{Mg}_{1/3}\text{Ta}_{2/3})\text{Zr}]\text{O}_3$ have the morphotropic phase boundary (MPB) between rhombohedral and tetragonal phases at $\text{PbZrO}_3 = 0.29 \sim 0.35$ [3]. The substitution of small amount of La^{3+} to $\text{Pb}_{1-3x/2}\text{La}_x[(\text{Mg}_{1/3}\text{Ta}_{2/3})_{0.66}\text{Zr}_{0.34}]\text{O}_3$ systems to decreases the temperature of normal phase transition. However, the origin of these properties is not completely clear.

In this study, $\text{Pb}[(\text{Mg}_{1/3}\text{Ta}_{2/3})_{0.66}\text{Zr}_{0.34}]\text{O}_3$ of the MPB composition range was selected for $\text{Pb}[(\text{Mg}_{1/3}\text{Ta}_{2/3})\text{Zr}]\text{O}_3$ system. The MPB composition of $\text{Pb}[(\text{Mg}_{1/3}\text{Ta}_{2/3})\text{Zr}]\text{O}_3$ is an intense research, but the pyroelectric properties of the material depend on small dopants, such as Bi, Sn, Mn and

La . $\text{Pb}[(\text{Mg}_{1/3}\text{Ta}_{2/3})_{0.66}\text{Zr}_{0.34}]\text{O}_3$ type polycrystalline systems were prepared by doping with La_2O_3 . The purpose of this study was to develop materials with superior dielectric properties, higher pyroelectric properties and higher pyroelectric FOM at room temperature.

II. Experimental results and Discussion

1. Experiments and observations

Solid solution samples of $\text{Pb}_{1-3x/2}\text{La}_x[(\text{Mg}_{1/3}\text{Ta}_{2/3})_{0.66}\text{Zr}_{0.34}]\text{O}_3$ of $x = 0 \sim 0.03$ mole% was prepared using the columbite precursor method [4]. The most common problem when manufacturing complex perovskites of Pb-type is to obtain pyrochlore phase (non-cubic) instead of perovskite. The samples were sintered at 1350°C for 4 hours at a heating rate of 300°C/h . All sintered pellets were analyzed using XRD. Prior to capacitance and pyroelectric current, the samples were poling by applying a DC electric field (20 kV/cm) for 30 minutes at room temperature. Dielectric constant is routinely calculated from the well-known equation follows $\epsilon = \frac{Cd}{\epsilon_0 A}$, where ϵ is the dielectric constant, C is the capacitance of sample, d is the thickness of sample, ϵ_0 is the permittivity of free space and A is the area of sample. Pyroelectric current was measured by the static Byer-Roundy method [5] when the samples was heated at a rate of 4°C/min through the phase transition region. Piezoelectric properties were measured using modified resonance/anti-resonance method and piezo- d_{33} meter [6].

X-ray powder diffraction was used to identify the perovskite phase produced. Fig. 1 shows the x-ray

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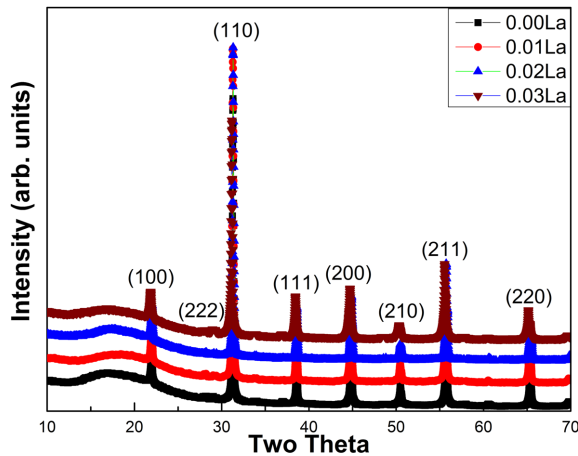


Figure 1. X-ray diffraction patterns of $\text{Pb}_{1-3x/2}\text{La}_x[(\text{Mg}_{1/3}\text{Ta}_{2/3})_{0.66}\text{Zr}_{0.34}]\text{O}_3$ systems.

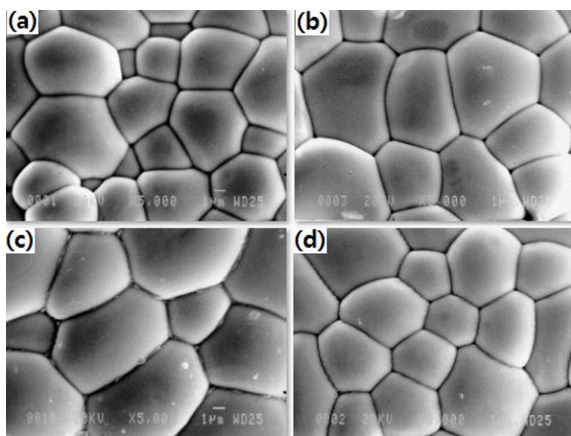


Figure 2. SEM images of $\text{Pb}_{1-3x/2}\text{La}_x[(\text{Mg}_{1/3}\text{Ta}_{2/3})_{0.66}\text{Zr}_{0.34}]\text{O}_3$ systems. (a) 0.00La, (b) 0.01La, (c) 0.02La and (d) 0.03La.

diffraction pattern of pure and La-modified $\text{Pb}_{1-3x/2}\text{La}_x[(\text{Mg}_{1/3}\text{Ta}_{2/3})_{0.66}\text{Zr}_{0.34}]\text{O}_3$ at room temperature. The x-ray diffraction pattern of $\text{Pb}_{1-3x/2}\text{La}_x[(\text{Mg}_{1/3}\text{Ta}_{2/3})_{0.66}\text{Zr}_{0.34}]\text{O}_3$ represents the perovskite (110) phase, and the pyrochlore (222) phase of the $\text{Pb}_{1-3x/2}\text{La}_x[(\text{Mg}_{1/3}\text{Ta}_{2/3})_{0.66}\text{Zr}_{0.34}]\text{O}_3$ system slightly increases as the La^{3+} content increases. This phenomenon is reported to be due to the substitution of La^{3+} ion in the Pb^{2+} -site of $\text{Pb}_{1-3x/2}\text{La}_x[(\text{Mg}_{1/3}\text{Ta}_{2/3})_{0.66}\text{Zr}_{0.34}]\text{O}_3$ ceramics as reported in $0.75(\text{Pb}_{1-3x/2}\text{La}_x)(\text{Mg}_{1/3}\text{Nb}_{2/3})\text{O}_3-0.25(\text{Pb}_{1-3x/2}\text{La}_x)\text{TiO}_3$ ceramics [7]. When doping La^{3+} to Pb^{2+} , charge imbalance must be considered using one of two methods. Pyrochlore phase is a non-cubic, isometric structure with excess tantalum, titanium, oxygen and its material properties, similar to the perovskite $\text{Pb}_{1-3x/2}\text{La}_x[(\text{Mg}_{1/3}\text{Ta}_{2/3})_{0.66}\text{Zr}_{0.34}]\text{O}_3$ compositions. Pyrochlore phases have also been considered detrimental to the dielectric properties of ferroelectric relaxor materials in addition to other factors such as impurities and inter-granular phases. There, the optimal heat treatment condition in this experiment was about 1350°C . Fig. 2 shows the micrograph of a scanning electron microscope for a composition $\text{Pb}_{1-3x/2}\text{La}_x[(\text{Mg}_{1/3}\text{Ta}_{2/3})_{0.66}\text{Zr}_{0.34}]\text{O}_3$ ($x=0, 0.02$). The grain size was partially increased by the addition of La_2O_3 but no

significant change was observed. The sintered sample has a grain size of 3.5 to 6.5 μm and a high density.

2. Dielectric properties

The temperature dependence of dielectric constant and loss at various frequencies (0.1 to 100 kHz) for the composition $\text{Pb}_{0.97}\text{La}_{0.02}[(\text{Mg}_{1/3}\text{Ta}_{2/3})_{0.66}\text{Zr}_{0.34}]\text{O}_3$ is shown in Fig. 3(a, b). This dielectric constant and dielectric loss data clearly show that the phase transition occurs from the paraelectric to the ferroelectric state. A relatively strong dielectric dispersion is observed at temperatures near the T_C , which is characteristic of the relaxors. Of course, all relaxors are known to be very inhomogeneous. The higher dielectric constant of the sample reflects good stoichiometry. The phase transition of relaxor ferroelectric affects the physical environments such as thermal agitation, grain size and grain boundaries. And the dielectric constant of relaxor decreases with increasing frequency and the phase transition temperature is biased towards higher temperature. The $\text{Pb}_{0.97}\text{La}_{0.02}[(\text{Mg}_{1/3}\text{Ta}_{2/3})_{0.66}\text{Zr}_{0.34}]\text{O}_3$ is known as a rhombohedral/tetragonal structure in a ferroelectric state. Fig. 3 shows that the phase transition mechanism of $\text{Pb}_{0.97}\text{La}_{0.02}[(\text{Mg}_{1/3}\text{Ta}_{2/3})_{0.66}\text{Zr}_{0.34}]\text{O}_3$ differs from the Curie-Weiss law. Relaxors can be separated into long-range ferroelectric orders from the T_C .

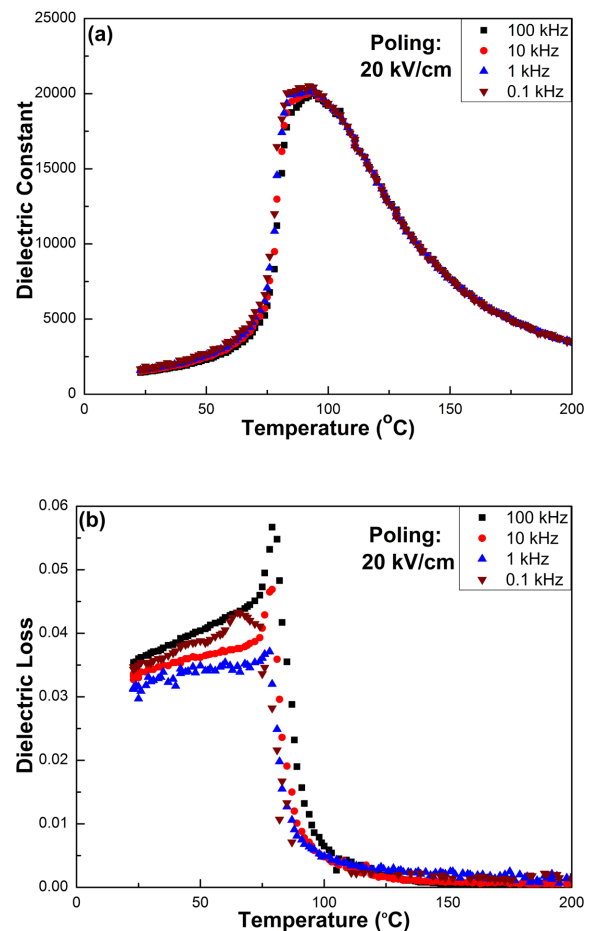


Figure 3. (a) Dielectric constant and (b) dielectric loss versus temperature of $\text{Pb}_{1-3x/2}\text{La}_x[(\text{Mg}_{1/3}\text{Ta}_{2/3})_{0.66}\text{Zr}_{0.34}]\text{O}_3$ at various frequencies.

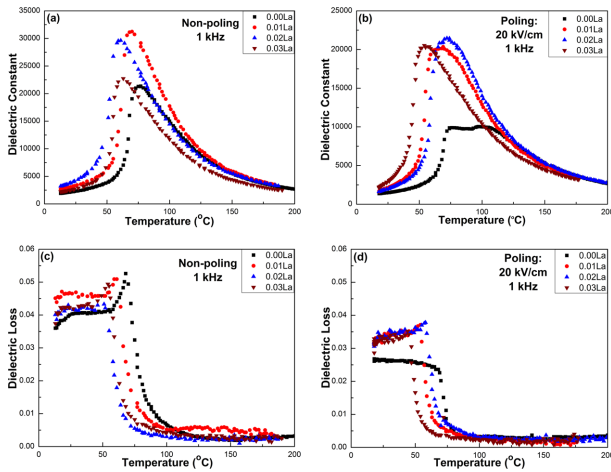


Figure 4 Dielectric properties of non-poling (a, c) and poling (b, d) $\text{Pb}_{1-3x/2}\text{La}_x[(\text{Mg}_{1/3}\text{Ta}_{2/3})_{0.66}\text{Zr}_{0.34}]\text{O}_3$ with varying La^{3+} concentrations.

The dielectric constant of relaxor near the Curie region is governed by the modified Curie-Weiss quadratic equation $\frac{1}{\epsilon} = \frac{1}{\epsilon_m} + \frac{(T-T_m)^\gamma}{2\epsilon_m Q^2}$, where ϵ is the dielectric constant, ϵ_m is the maximum dielectric constant, T_m is the dielectric constant maxima temperature, δ is the diffuseness parameter and γ is the critical exponent. In this experiment, the frequency dispersion and diffusion of the transition increases as the amount of La_2O_3 in the composition increases. This phenomena is similar to that of the $\text{Pb}_{1-x}\text{La}_x[(\text{Mg}_{1+x/3}\text{Nb}_{2-x/3})_{0.65}\text{Ti}_{0.35(1-x)/4}]\text{O}_3$ with the same Pb-type perovskite [8].

Fig. 4(a, b, c, d) shows the temperature dependence of the dielectric constant and loss of $\text{Pb}_{1-3x/2}\text{La}_x[(\text{Mg}_{1/3}\text{Ta}_{2/3})_{0.66}\text{Zr}_{0.34}]\text{O}_3$ at 1 kHz in a series of different La^{3+} concentrations, compare the non-poling (Fig. 4(a, c)) and poling (Fig. 4(b, d)) samples. Most complex perovskite ferroelectrics have several advantages. Transitions generally exhibit large thermal hysteresis. Well-sintered ceramics have high dielectric constant but low dielectric loss. When the soft doping ions enter the lattice structure, the effect of the modified La_2O_3 is due to attributed to the certain of Pb^{2+} vacancies in the $\text{A}(\text{B}'\text{B}'')\text{O}_3$ perovskite lattice. Since the Pb^{2+} ions occupy the A-site of the perovskite lattice, soft doping ions play a role in generating A-site vacancies. Therefore, since the valence of the crystal ion is higher than that the valence of the Pb^{2+} ion, an extra positive charge is introduced into the lattice. When two A-sites are occupied by two cations having a valence of +3, Pb vacancies are generated in the lattice to maintain electrical neutrality [9]. In other words, it is considered that A-site vacancy occurs due to the successive reaction of $\text{Mg}_{1+x/3}\text{Ta}_{2-x/3}\text{O}_{4-x/2}$ and $\text{La}_x\text{Pb}_{1-x}(\text{Mg}_{1+x/3}\text{Ta}_{2-x/3})\text{O}_3$ during the fabrication of $\text{Pb}_{1-3x/2}\text{La}_x[(\text{Mg}_{1/3}\text{Ta}_{2/3})_{0.66}\text{Zr}_{0.34}]\text{O}_3$. Therefore, the dielectric constant of $\text{Pb}_{1-3x/2}\text{La}_x[(\text{Mg}_{1/3}\text{Ta}_{2/3})_{0.66}\text{Zr}_{0.34}]\text{O}_3$ was the highest. It can be related to possible local variations of the La^{3+} concentration. In Fig. 4(a, c), the maximum value of the dielectric constant increases rapidly at the beginning and decreases as the La^{3+} content increases, but the frequency dispersion behavior is still observed. The maximum value of

the dielectric constant is slightly increased by addition of La^{3+} up to 2 mole%, but decreased by further addition of La^{3+} . The dielectric loss increased initially and slightly decreased with further addition of La^{3+} . The increase in the amount of La_2O_3 strongly affects the T_C , which is approximately linearly decreased. This is attributed to the change in c/a in the crystal lattice of the $\text{Pb}_{1-3x/2}\text{La}_x[(\text{Mg}_{1/3}\text{Ta}_{2/3})_{0.66}\text{Zr}_{0.34}]\text{O}_3$ compositions. It has been found that the behavior of the additive effect on dielectric properties is very similar to that of the $\text{Pb}[(\text{Mg}_{1/3}\text{Nb}_{2/3})\text{Zr}]\text{O}_3$ composition. La^{3+} doping caused a downward shift of $T_C \sim 12^\circ\text{C}/\text{mole}\%$ in $\text{Pb}[(\text{Mg}_{1/3}\text{Ta}_{2/3})\text{Zr}]\text{O}_3$ polycrystalline ceramics.

3. Pyroelectric properties

The pyroelectric coefficient is routinely calculated from well-known equation follows $i_p = A_p \frac{dT}{dt}$, where i_p is the pyroelectric current, A is the area of sample, p is the pyroelectric coefficient, and dT/dt is the heating rate. Fig. 5 and 6 show the pyroelectric coefficient and the spontaneous polarization for the composition of $\text{Pb}_{1-3x/2}\text{La}_x[(\text{Mg}_{1/3}\text{Ta}_{2/3})_{0.66}\text{Zr}_{0.34}]\text{O}_3$ for a series of different La^{3+} concentrations. The peak of the pyroelectric coefficient initially increases and rapidly decreases with increasing temperature. The composition with maximum pyroelectric coefficient and spontaneous polarization may be similar to the composition observed in the dielectric constant vs. temperature study. The pyroelectric coefficient increases with the mole% of La^{3+} , but if La^{3+} is larger than ~ 0.03 , the pyroelectric coefficient decreases. The activation barrier of Pb-perovskite is dictated by the strength of the Pb-O-B'' bonds, which must be destroyed to relax the material. This can be achieved by substitution Pb^{2+} cations at the A-site using large B' cations and pulling the Pb^{2+} cations at the small B'' cations [10]. In the phenomenological theory of ferroelectricity, there is an energy barrier between the unpolarized state and the polarized state at the first order phase transition temperature. At the atomic level, most ferroelectric perovskites ferroelectrically activated B cations occupy an off-center position above the Curie temperature.

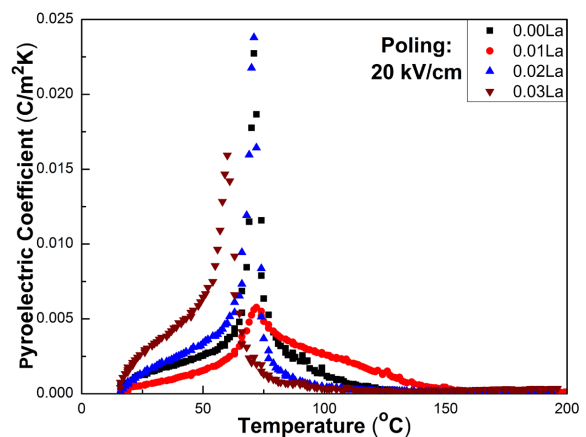


Figure 5 Pyroelectric coefficient of $\text{Pb}_{1-3x/2}\text{La}_x[(\text{Mg}_{1/3}\text{Ta}_{2/3})_{0.66}\text{Zr}_{0.34}]\text{O}_3$ with varying La^{3+} concentrations.

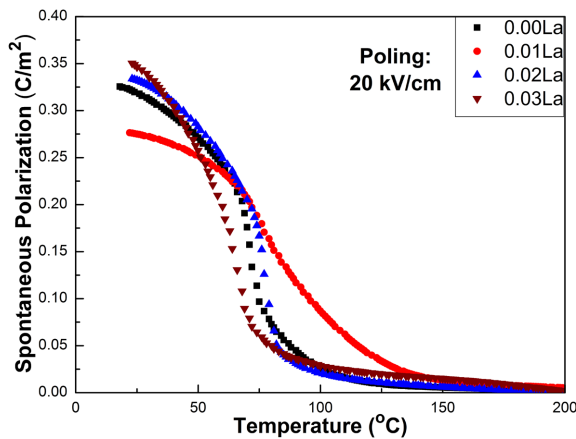


Figure 6. Spontaneous polarization of $\text{Pb}_{1-3x/2}\text{La}_x[(\text{Mg}_{1/3}\text{Ta}_{2/3})_{0.66}\text{Zr}_{0.34}]\text{O}_3$ with varying La^{3+} concentrations.

In this regard, the dielectric response of the relaxor is similar to that exhibited by many amorphous materials, including many of the weak/strong bonds, isolated electron pairs, etc., which are thought to cause low-lying excitations of correlated polarity. The orientation or polarization fluctuation of the dipole in the cluster affects not only the position of the adjacent cation but also the polarization of adjacent region [11,12,13].

Fig. 6 shows the spontaneous polarization decreases gradually with increasing temperature, but does not disappear at the transition temperature of $\text{Pb}_{1-3x/2}\text{La}_x[(\text{Mg}_{1/3}\text{Ta}_{2/3})_{0.66}\text{Zr}_{0.34}]\text{O}_3$. This is typical of a relaxor ferroelectric. Spontaneous polarization indicates that another dipole is destroyed when the temperature increases. As the La^{3+} concentration increases, the peak temperature decreases rapidly. The spontaneous polarization showed a value of $0.27\sim 0.35\text{ C/m}^2$ in the composition added to La^{3+} . This value is excellent as the the spontaneous polarization value of ferroelectric relaxor. In this composition, La^{3+} ions are replaced by Pb^{2+} ions on the A-site sub-lattice. Since La^{3+} is trivalent and Pb^{2+} is divalent, La^{3+} behaves as a donor dopant. These phenomena can result in the distribution of La^{3+} (1.03 pm) and Pb^{2+} (1.19 pm) ions with different ionic radii at the Pb-site. But they cannot be fully explained at this time. The addition of La^{3+} ions to $\text{Pb}_{1-3x/2}\text{La}_x[(\text{Mg}_{1/3}\text{Ta}_{2/3})_{0.66}\text{Zr}_{0.34}]\text{O}_3$ resulted in cation vacancies in the lattice for retention of electronegativity, lowering the peak temperature and increasing the pyroelectric coefficient at about room temperature. With the substitution of La^{3+} , the peak temperature of $\text{Pb}_{1-3x/2}\text{La}_x[(\text{Mg}_{1/3}\text{Ta}_{2/3})_{0.66}\text{Zr}_{0.34}]\text{O}_3$ can be adjusted to suit room temperature pyroelectric applications. Recently, a pyroelectric effect of a ferroelectric has attracted attention for application to infrared detectors. The properties required for such large pyroelectric coefficient, low dielectric constant and $\tan\delta$ values. The pyroelectric response of the material is generally related to the application of the device by the FOMs $F_V = \frac{p}{c_v \epsilon_r \epsilon_0}$ and $F_D = \frac{p}{c_v \sqrt{\epsilon_r \epsilon_0 \tan\delta}}$, where C_V is the volume specific heat, ϵ_r is

the dielectric constant, ϵ_0 is the permittivity of free space, and $\tan\delta$ is the dielectric loss. The pyroelectric FOMs ($F_V \sim 0.012\sim 0.035\text{ m}^2/\text{C}$, $F_D \sim 0.15 \times 10^{-4}\sim 0.52 \times 10^{-4}\text{ Pa}^{-1/2}$) of these ceramics is similar to the published values for Pb-perovskites [14,15]. The piezoelectric constant ($d_{33} \sim 490\text{ pC/N}$) and electromechanical coupling factor ($k_p \sim 0.35$) are obtained in the $\text{Pb}_{0.97}\text{La}_{0.02}[(\text{Mg}_{1/3}\text{Ta}_{2/3})_{0.66}\text{Zr}_{0.34}]\text{O}_3$ composition with 0.02 mole% of La.

III. Conclusions

Most compositions show diffuse phase transition with strong frequency dispersion. The frequency dispersion and diffusion of the transition increase as the amount of La_2O_3 in the composition increases. The maximum values of the dielectric constant and the pyroelectric coefficient are slightly increased by addition of La^{3+} up to 2 mole% and then decreased by further addition of La^{3+} ion. La^{3+} ion doping resulted in a $T_C \sim 12^\circ\text{C}/\text{mole}\%$ downward shift in the $\text{Pb}_{1-3x/2}\text{La}_x[(\text{Mg}_{1/3}\text{Ta}_{2/3})_{0.66}\text{Zr}_{0.34}]\text{O}_3$ polycrystalline solid solution. The composition near $\text{Pb}_{0.97}\text{La}_{0.02}[(\text{Mg}_{1/3}\text{Ta}_{2/3})_{0.66}\text{Zr}_{0.34}]\text{O}_3$ was confirmed to have a high pyroelectric FOM near room temperature. The pyroelectric FOM of these ceramics at room temperature is similar to the Pb-perovskites. The spontaneous polarization showed a value of $\sim 0.35\text{ C/m}^2$ in the composition added to 0.03 mole% of La^{3+} . In the $\text{Pb}_{1-3x/2}\text{La}_x[(\text{Mg}_{1/3}\text{Ta}_{2/3})_{0.66}\text{Zr}_{0.34}]\text{O}_3$ system, the piezoelectric constant ($d_{33} = 350\sim 490\text{ pC/N}$) and electromechanical coupling factor ($k_p = 0.25\sim 0.35$) are obtained.

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