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The study of dielectric, pyroelectric and piezoelectric properties on hot pressed PZT-PMN systems

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Hot uniaxial pressing technique has been adopted for the densification of PZT-PMN system with an aim to yield dense ceramics and to lower the sintering temperature and time for achieving better and reproducible electronic properties. The ceramics having >97% theoretical density and micron size grains are investigated for their dielectric, pyroelectric and piezoelectric properties. The effect of Li and Mn addition has also been studied. Copyright 2012 Author(s). This article is distributed under a Creative Commons Attribution 3.0 Unported License. [<http://dx.doi.org/10.1063/1.4769889>]

I. INTRODUCTION

Lead based perovskites have been extensively studied for their excellent dielectric and piezoelectric properties. PMN is known for its high and broad range of dielectric constant with low loss^{1,2} and non-hysteretic behavior³⁻⁵ making them suitable for multilayer capacitors, sensors and actuators. It also exhibits relatively low electromechanical coupling as compared to PZT but single phase PMN ceramics is difficult to synthesize. PZT ceramics are characterized by high electromechanical coupling but possess high Curie temperature and have high hysteretic characteristics,⁵ which are not suited to device applications. Therefore it is preferred to form a solid solution of PZT and PMN so as to combine the complementary features of PZT and PMN systems.⁶⁻¹³ The primary intention is not only to enhance and optimize the material properties but also to improve the sinterability of the system.

It is known that these lead based ceramics generally requires high sintering temperature which results in compositional fluctuation due to the evaporation of PbO thereby deteriorating the properties like electromechanical, piezoelectric, dielectric and also causing the environmental pollution. Hence, low temperature sintering is an important parameter to improve the piezoelectric properties, reliability and reproducibility of these lead based perovskites. The low temperature fabrication of these perovskite ceramics have been realized by various methods like (i) using additives which promotes liquid phase sintering (ii) hot pressing and (iii) by the use of ultra fine particles prepared by chemical methods.

The reduction of sintering temperature of these lead based ceramics was attempted through the addition of numerous metal oxide additives and has been reported on several occasions.¹⁴ Several sintering aids that have been tried in the literature include CuO, PbO, B₂O₃, Bi₂O₃, LiBiO₂, BiFeO₃, Li₂CO₃ etc¹⁵⁻²¹ which were found to either reduce the piezoelectric properties or the sintering temperature still could not be lowered much.

Li₂CO₃ is one of the most used additives for this purpose in the past. It was observed by You and Koh that the sintering temperature was lowered from 1623 K to 1173 K when Li₂CO₃ was added to (Ba, Sr) TiO₃ system.²² Further, Yoo *et al.* reported the densification of PbZr_{0.52}Ti_{0.48}O₃ ceramics at 1213 K with Li₂CO₃ as a sintering aid and the piezoelectric properties of these ceramics were also

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found to be superior to that of parent system.⁶ There have been numerous studies on the Mn doping in the PZT system as well.^{23–27} In our earlier work the addition of Li and Mn in the PZT based system led in the enhancement of material properties.²⁸ It has been observed that Li and Mn addition in the PZT-PZN system results in better sinterability, densification and dielectric properties.²⁸

In this work, hot uniaxial pressing of the ceramics was attempted with intentions (i) to avoid the volatilization of PbO (ii) to densify the piezoelectric ceramics. In our earlier work, we adopted low temperature calcination route to stabilize perovskite phase in PMN-PT ceramics.²⁹ This method yielded in fine particles which made sintering mechanism easier. The aim of this study is to produce high grade piezoelectric PZT-PMN ceramics with high material properties. For this purpose, we adopted low temperature calcination route followed by uniaxial hot pressing of ceramics. Further, the effects of additives like Li and Mn on the material properties such as dielectric, piezoelectric, pyroelectric were also investigated.

II. EXPERIMENTAL DETAILS

PZT-PMN ceramics with nominal composition $\text{Pb}[(\text{Zr}_y\text{Ti}_{1-y})_{1-x}(\text{Mg}_{1/3}\text{Nb}_{2/3})_x]\text{O}_3$ ($x = 0.1, 0.2$) were prepared by two-stage synthesis method. The starting powders were calcined at 973 K for an hour. The calcined powders were then hot pressed at ~ 1273 K at 28MPa in vacuum atmosphere for 15 min. The hot pressed pellets were annealed at 1173 K. For the addition of Li and Mn in the system, 0.2 wt% of Li_2CO_3 and 0.15 wt% Mn were added in solution form to the nominal composition after calcination stage and was preheated at 773 K to remove all the volatile materials prior to hot pressing. X-ray diffraction data were collected on the ceramic samples at normal scanning rate of $1^\circ/\text{minute}$, with a step size of 0.02° using Phillips PW1050/37 diffractometer with $\text{Cu K}\alpha$ radiation.

The density of all the hot pressed annealed samples was determined using modified three weight Archimedes' principle.

The pellets were gold coated and then silver wire electrode was attached with silver epoxy. Specimens for piezoelectric and pyroelectric measurements were poled close to 550 K by applying a dc electric field of 2 kV cm^{-1} and cooling down to room temperature in silicone oil bath. Piezoelectric properties were measured by the resonance –anti-resonance method on the basis of IRE Standards³⁰ using an impedance analyzer (HP4194A). The pyroelectric coefficient, p , and spontaneous polarization, P_s , were measured by the Byer and Roundy method³¹ with the temperature rate of 4 Kmin^{-1} using Keithley 610 C electrometer. The sample holder was evacuated and filled with helium gas to ensure that the ambient humidity did not influence the measurement. The variation of pyroelectric coefficient, p_i , and spontaneous polarization, P_s with the temperature has been studied for the synthesized samples.

The pyroelectric coefficient p_i is calculated using the equation as mentioned below :

$$p_i(T) = \frac{I}{A(dT/dt)} \quad (1)$$

where dT/dt is the rate of temperature variation

I is the pyroelectric current

A is the surface area of the sample

III. RESULTS

A. Phase Formation

The samples were calcined at 973 K to bypasses the formation of $\text{Pb}_3\text{Nb}_4\text{O}_{13}$ type pyrochlore which is difficult to eliminate in the sintering process.²⁹ The x-ray diffraction patterns of the calcined powders of all samples showed the presence of desired pyrochlore $\text{Pb}_{2.31}\text{Nb}_2\text{O}_{7.31}$ phase (JCPDS No. -43-0961), which is B-site deficient, along with the traces of PbO and perovskite phase.²⁹ The hot pressed pellets obtained after annealing yielded pure perovskite phase for all the samples. No

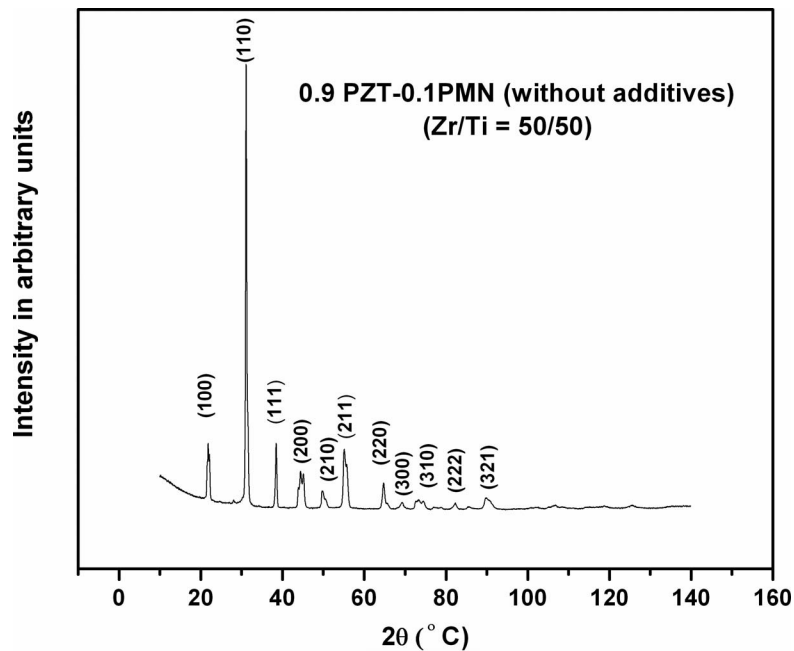


FIG. 1. A typical XRD pattern of the sintered ceramic of 0.9 PZT-0.1PMN composition without any additives showing the pure perovskite phase formation.

other secondary phase was observed within the detection limit of x-ray. The x-ray diffraction pattern of the typical perovskite phase has been depicted in Fig. 1.

In our previous work, it has been found that with the introduction of PZN and PMN to the PZT system, the morphotropic phase boundary (MPB) shifts towards PbZrO_3 .^{28,32} Further it was found that the system can be restored to the MPB region by adjusting the Zr/Ti ratio in the system.³² It has been noted that the compositions close to the MPB show extremum material properties such as piezoelectric, electromechanical coupling coefficient. Hence, the materials are synthesized close to the MPB region. So, in this work the Zr/Ti ratio was chosen such that the system retains the MPB for 0.9PZT-0.1PMN and 0.8PZT-0.2PMN systems. The ratio of Zr/Ti was found to be 50/50 for $x = 0.1$ and 42/58 for $x = 0.2$.²⁸ The Li and Mn additives were also tried for both the parent compositions.

B. Density and SEM studies

The density of all the hot pressed ceramics have been found to be >97% of the theoretical density. The porosity of all the samples has also been calculated to be less than 5%. The typical scanning electron micrographs (SEM) have been shown in Fig. 2. It can be inferred from Fig. 2 that the hot uniaxial pressing resulted in highly dense piezoelectric ceramics with finer and uniform microstructure. The grain size calculated was found to be less than $1 \mu\text{m}$ and was attributed to hot pressing. Here the low temperature and short duration helped in restricting the grain growth of the samples.

C. Dielectric studies

The dielectric constant and loss of the materials of different compositions were determined with variation in the temperature. The dielectric permittivities of all the compositions at room temperature as well as transition temperature T_c with corresponding dielectric losses (1 kHz) have been tabulated in Table I. The dielectric properties are found to be superior than reported in literature as obtained through solid state synthesis approach.^{33–36} The reason for the improvement in the dielectric properties for the PZT-PMN systems may be explained on the basis of high density,

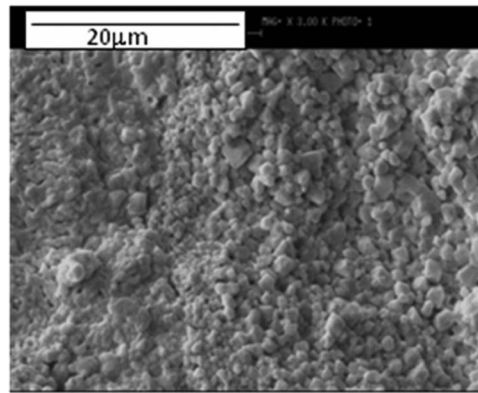


FIG. 2. A typical SEM image of the hot pressed ceramic.

TABLE I. Summarized dielectric properties of various PZT-PMN (1 kHz) compositions.

System	ϵ_{RT}	δ_{RT}	$T_C(K)$	$\epsilon_{max}(T_C)$	δ_{T_C}
0.9PZT-0.1PMN	1408	0.039	651	16476	0.07
0.9PZT-0.1PMN with 0.2 wt%Mn	1469	0.04	603	17725	0.05
0.9PZT-0.1PMN with 0.15 wt%Li	1598	0.02	624	17915	0.05
0.8PZT-0.2PMN	1056	0.04	640	9638	0.09
0.8PZT-0.2PMN with 0.2 wt%Mn	1154	0.03	597	12218	0.06
0.8PZT-0.2PMN with 0.15 wt%Li	1219	0.016	608	13397	0.06

better homogeneity and fine microstructure resulting from the hot pressing technique adopted for the synthesis of these materials. Further, it is generally observed that the PMN rich and PZT rich compositions show relatively large dielectric constant.³⁷

The transition temperature of 0.9PZT-0.1PMN is found to be 651 K which reduced to 640 K for 0.8PZT-0.2PMN. It is known that transition temperature of PMN is below room temperature i.e 265 K to 258 K. Hence, the transition temperature for the investigated compositions was found to decrease with the increase in content of PMN in the system. Li and Mn addition too were found to lower the transition temperature of the PZT-PMN system. However the reduction in the transition temperature was found to be more in case of Mn added composition than Li added system. The transition temperature for Mn added system reduced to 603 K and 597 K for 0.9PZT-0.1PMN and 0.8PZT-0.2PMN compositions compared to 624 K and 608 K for Li added compositions.

It is observed that Li and Mn addition led to the increase in the dielectric property. The maximum dielectric permittivity at transition temperature with Li addition increased from 16476 to 17915 as compared to parent 0.9PZT-0.1PMN. Similarly for the composition 0.8PZT-0.2PMN, the maximum dielectric permittivity was observed as 13397 compared to 9638 of parent composition. A decrease of the dielectric losses was also found for both additives.

D. Pyroelectric studies

The variation of pyroelectric coefficient p_i as a function of temperature has been depicted in Fig. 3. The maximum pyroelectric coefficient p_i is found to be $0.148 \mu\text{Ccm}^{-2}/\text{K}$ for 0.8PZT-0.2PMN compared to $0.165 \mu\text{Ccm}^{-2}/\text{K}$ for Mn added and $0.194 \mu\text{Ccm}^{-2}/\text{K}$ for Li added compositions. Similarly the parent composition of 0.9PZT-0.1PMN revealed the maximum p_i of $0.237 \mu\text{Ccm}^{-2}/\text{K}$ compared to $0.249 \mu\text{Ccm}^{-2}/\text{K}$ for Mn and $0.280 \mu\text{Ccm}^{-2}/\text{K}$ for Li added compositions. Hence, it has been proved that Li and Mn added compositions yielded better pyroelectric activity than the parent compositions.

The trend in the phase transitions in the system was found to be complementing the dielectric studies. The spontaneous polarization P_s was calculated by integrating the value of p_i vs T and

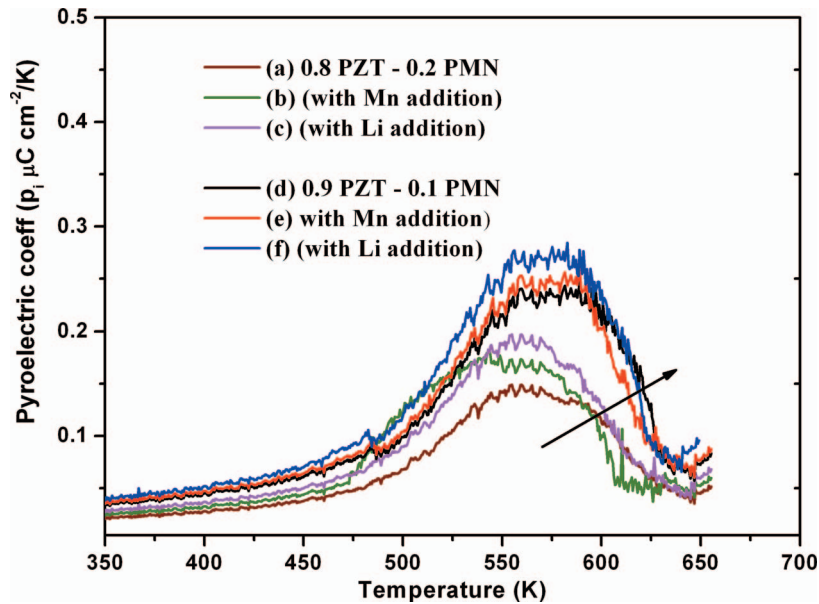


FIG. 3. The variation of pyroelectric coefficient with temperature for various PZT-PMN compositions.

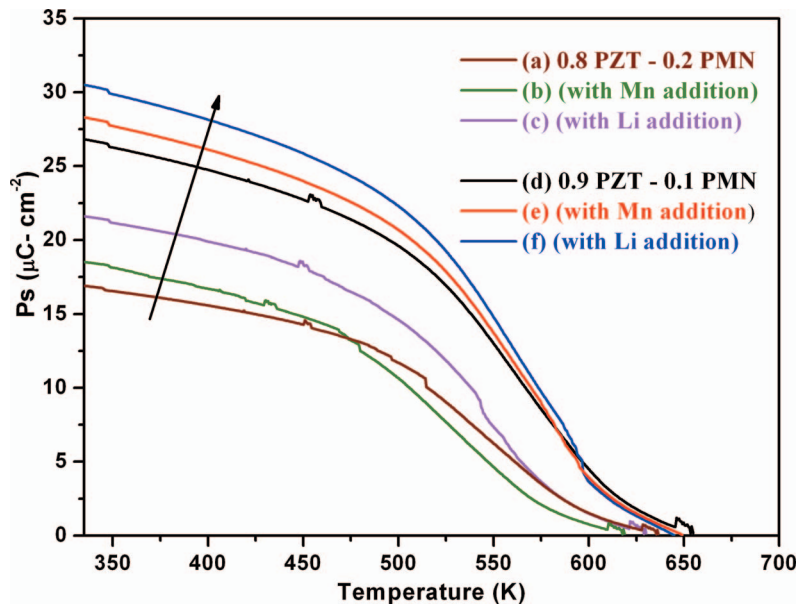


FIG. 4. The variation of spontaneous polarization with temperature for various compositions of PZT-PMN.

has been plotted as a function of temperature in Fig. 4. The value of P_s obtained was maximum ($30 \mu\text{Ccm}^{-2}$) for Li added 0.9PZT-0.1PMN composition whereas it was reported to be $18 \mu\text{Ccm}^{-2}$ for parent composition of 0.8PZT-0.2PMN. Further, it can be seen from the figure that the polarization is lost above the transition temperature T_c .

E. Piezoelectric studies

An investigation of the temperature dependence of the piezoelectric properties and electromechanical coupling factor has been carried out on PZT-PMN system for various compositions to

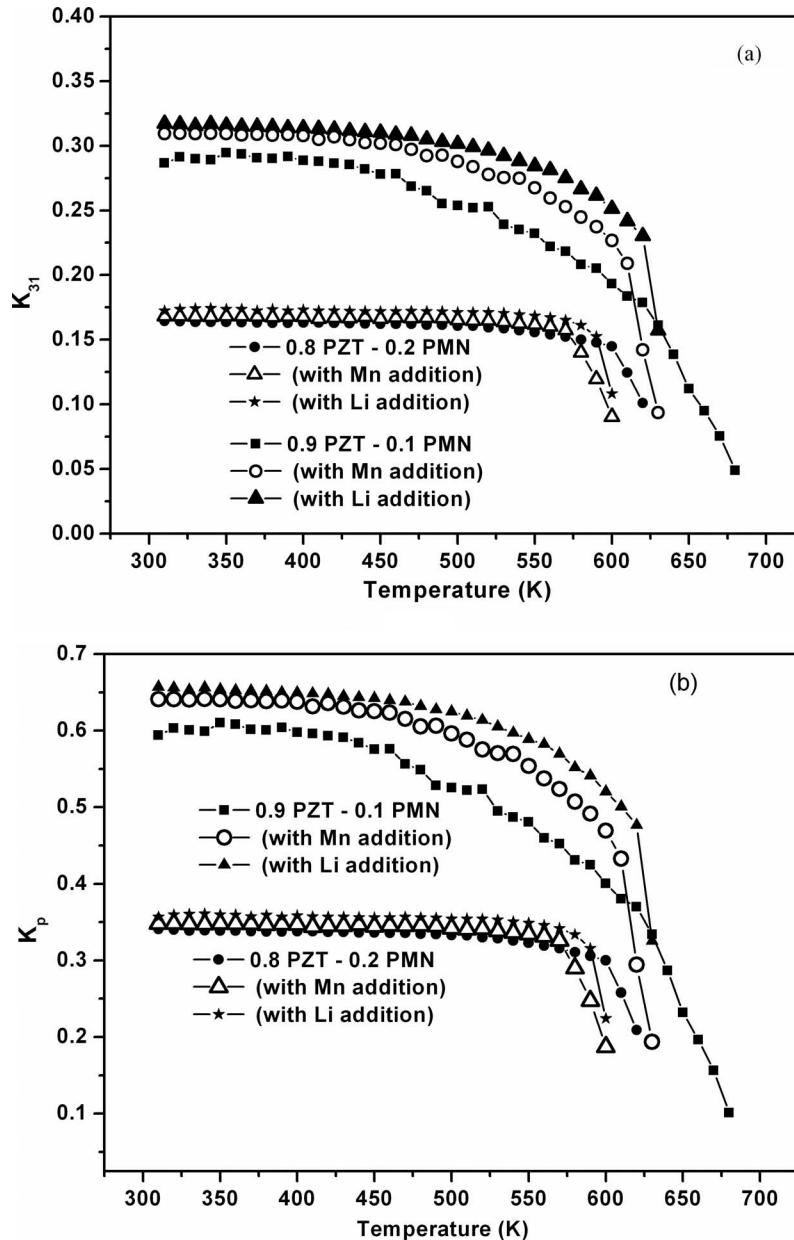


FIG. 5. Temperature dependence of (a) radial coupling coefficient k_{31} and (b) planar coupling coefficient k_p for different compositions of PZT-PMN.

evaluate the performance of the material for device applications. These properties were measured from room temperature to ~ 675 K.

The planar coupling coefficient denoted by k_p and radial coupling factor, k_{31} are generally used as measures for piezoelectric response of thin disks. Their value depends very much on the density, and porosity of the sample. Furthermore, it varies with chemical composition. The variation in the planar coupling coefficient, k_p , and radial coupling coefficient, k_{31} , for various compositions of PZT-PMN with the temperature has been plotted in Figs. 5(a) and 5(b). It can be seen that it almost remains constant for all the compositions till the depoling in the material begins. Hence it can be stated as the system presents an excellent thermal stability, in the temperature range investigated till the depoling in the material starts and the coupling coefficients values initiates to decrease from its maximum attained value.

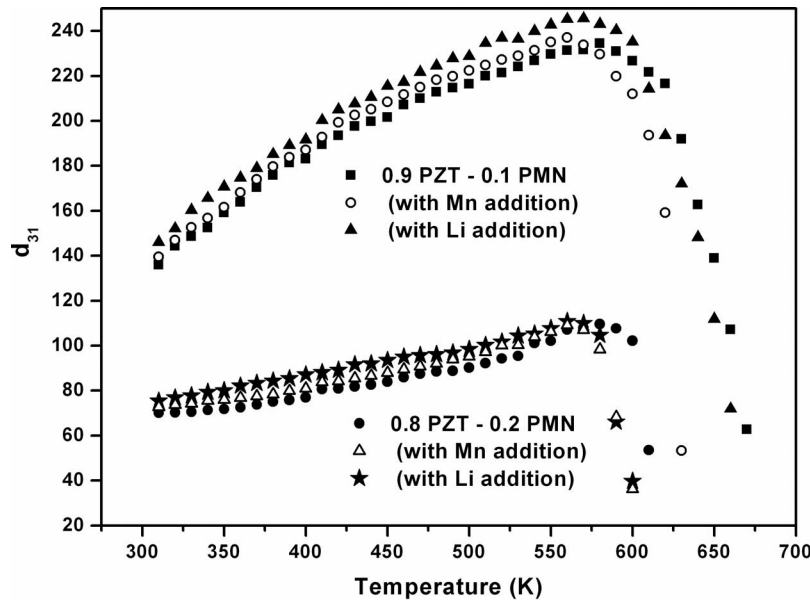


FIG. 6. The variation of piezoelectric coefficient d_{31} for various compositions of PZT-PMN with the temperature

TABLE II. Summarized piezoelectric, pyroelectric and electromechanical properties of various PZT-PMN compositions at room temperature.

System	d_{31} (pC/N)	K_p	K_{31}	P_s ($\mu\text{C}/\text{cm}^2$)	p_i ($\text{nC}/\text{cm}^2\text{-K}$)
0.9PZT-0.1PMN	136	0.593	0.287	29	8.09
0.9PZT-0.1PMN with 0.2 wt%Mn	140	0.641	0.310	31	8.32
0.9PZT-0.1PMN with 0.15 wt%Li	146	0.657	0.317	33	8.52
0.8PZT-0.2PMN	70	0.341	0.165	19	5.01
0.8PZT-0.2PMN with 0.2 wt%Mn	73	0.348	0.168	21	5.83
0.8PZT-0.2PMN with 0.15 wt%Li	75	0.357	0.172	24	6.63

The maximum coupling coefficient k_p , k_{31} of 0.657 and 0.317 was observed for Li added 0.9PZT-0.1 PMN composition compared to 0.593 and 0.287 of parent composition. Mn added composition too showed enhanced values of k_p and k_{31} than the parent composition.

The lateral strain coefficient, denoted by d_{31} was calculated for different temperature and has been plotted in Fig. 6. The same trend in the piezoelectric coefficient was observed for different composition of PZT-PMN system. Li and Mn added composition showed marked improvement in the values of d_{31} . The maximum value obtained was 146pC/N for Li added 0.9PZT-0.1PMN composition compared to 136pC/N of parent composition. It reaches a maximum of 246pC/N with the rise in the temperature. The increase in the value of piezoelectric coefficient is expected with the temperature as there exists a direct relation between the dielectric permittivity and the piezoelectric coefficient. The dielectric permittivity increases with the increase in the temperature till the transition temperature is reached. Hence, there is an observed variation in the value of d_{31} with temperature. However, there is larger temperature dependence seen for the composition of 0.9PZT-0.1PMN compared to 0.8PZT-0.2PMN. This kind of variation in temperature dependence was observed for PZT system near the morphotropic phase boundary and it was argued that the compositions close to MPB is seen to have larger temperature dependence of piezoelectric coefficients compared to the compositions far away from the MPB region.

The electromechanical, piezoelectric and pyroelectric properties at room temperature of various PZT-PMN compositions have been summarized in Table II

IV. CONCLUSIONS

The hot pressing technique (adopting low temperature calcination) was effective in yielding highly dense piezo-ceramics with fine, homogeneous and uniform microstructure which resulted in excellent material properties of the system. The dielectric, pyroelectric and piezoelectric behavior were studied on the various PZT-PMN composition. The transition temperature obtained from the dielectric, pyroelectric and piezoelectric studies for system under investigation complemented each other. The solid solutions of Li and Mn added PZT-PMN exhibit higher pyroelectric activity than the parent compositions.

The temperature dependence of the electrical and electromechanical properties of PMN-PZT system was also investigated. The temperature dependent study of the electromechanical property indicates that the system is thermally stable. The result obtained in this study suggests that the Li and Mn additions to the parent composition of PZT-PMN led to an overall improvement in the material properties.

The complementary use of hot pressing and cationic additives is thus a way to improve the materials properties at the expense of a lower thermal budget.

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