

Article

Optimizing Sintering Temperature for Enhanced Piezoelectric Performance in PMT-PNT-PZT Ceramics

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Abstract

The 0.006Pb(Mn_{1/3}Ta_{2/3})O₃-0.114Pb(Ni_{1/3}Ta_{2/3})O₃-0.43PbZrO₃-0.45PbTiO₃ lead-based ceramics (PMT-PNT-PZT) were synthesized via the solid-state reaction at different sintering temperatures to study their effects on phase structure, microstructure, and electrical properties. The maximum mechanical quality factor (Q_m) and relative permittivity (ϵ_r) were achieved at the sintering temperature of 1200 °C. The piezoelectric constant d_{33} of 400 pC/N was obtained at 1180 °C, which is attributed to the high grain density and the significant contribution from the remanent polarization and permittivity product ($P_r\epsilon_r = 39,115 \mu\text{C}/\text{cm}^2$). Compared with commercial PZT4 ceramics, the present composition sintered at 1180 °C exhibits an optimal balance between d_{33} and Q_m , together with the superior figure of merit ($\text{FOM} = 2.04 \times 10^5 \text{ pC}/\text{N}$). Furthermore, it demonstrates excellent temperature stability in electromechanical coupling performance.

Keywords: PZT-based ceramics; sintering temperature; piezoelectric performance; figure of merit

1. Introduction

Piezoelectric materials enable efficient interconversion between electrical and mechanical energy, making them essential functional components in transducers, actuators, sensors, and energy harvesters [1–6]. Among them, lead-based ceramics such as PZT have been widely adopted owing to their excellent electrical properties [7–14]. To further enhance piezoelectric performance, lead-based relaxor ceramics have been developed, utilizing their unique microstructures to attain improved functional behavior.

PZT-based solid-solution piezoelectric ceramics are often modified or doped for specific applications. The introduction of rare earth elements Sm³⁺ and Eu³⁺ in PMN-PT ceramics has proven effective in improving dielectric properties [15–17]. Similarly, the incorporation of Mn and Fe has been reported to effectively enhance the mechanical quality factor [18–21]. Another strategy involves forming relaxor solid solutions, such as Pb(Ni_{1/3}Nb_{2/3})O₃-PbZrO₃-PbTiO₃ [22], Pb(Sn_{1/3}Nb_{2/3})O₃-Pb(Zn_{1/3}Nb_{2/3})O₃-Pb(Zr,TiO₃) [23], and Pb(Sc_{1/2}Nb_{1/2})O₃-PbTiO₃ [24] to enhance piezoelectric performance. While many studies on PZT-based ceramics focus on increasing the piezoelectric constant d_{33} or the planar electromechanical coupling factor k_p , only a limited number aim to achieve a balanced improvement in both d_{33} and Q_m . This balance is crucial for high-power applications, as d_{33} influences the sensitivity of ultrasonic transducers, while Q_m is associated



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with heat generation under high-drive conditions [25,26]. Therefore, the coordination of d_{33} and Q_m is of great significance for high-power application materials. [27]. In this work, $0.006\text{Pb}(\text{Mn}_{1/3}\text{Ta}_{2/3})\text{O}_3\text{-}0.114\text{Pb}(\text{Ni}_{1/3}\text{Ta}_{2/3})\text{O}_3\text{-}0.43\text{PbZrO}_3\text{-}0.45\text{PbTiO}_3$ (PMT-PNT-PZ-PT) is designed to simultaneously enhance d_{33} and Q_m , showing promising potential for high-power device applications.

Sintering temperature plays a critical role in determining the properties of piezoelectric ceramics. MnO_2 -doped PZT-PZN ceramics were meticulously prepared at a high temperature of 900°C , exhibiting an impressive d_{33} of 330 pC/N^{-1} and a remarkable Q_m of 1000 [28]. The $\text{Pb}(\text{Mn}_{1/3}\text{Nb}_{2/3})\text{O}_3\text{-Pb}(\text{Ni}_{1/3}\text{Nb}_{2/3})\text{O}_3\text{-Pb}(\text{Zr}_{0.50}\text{Ti}_{0.50})\text{O}_3$ (PMN-PNN-PZT) ceramics were sintered at 900°C , exhibiting a d_{33} of 346 pC/N^{-1} and a Q_m of 1130 [29]. The PMN-PZT- Li_2CO_3 ceramics were sintered at a temperature of 940°C , exhibiting a high quality factor Q_m (2264), a high Curie temperature T_c (317°C) and an impressive dielectric constant (1216) [30]. The PSNT-Mn with LiBiO_2 ceramics were sintered at 950°C , resulting in $d_{33} = 340\text{ pC/N}^{-1}$, $Q_m = 800$, $T_c = 263^\circ\text{C}$ [31]. PNN-PZN-PMN-PZ-PT ceramics were sintered at 950°C , exhibit excellent piezoelectric properties $d_{33}^* = 503\text{ pmV}^{-1}$, $Q_m = 471$ [32]. By exploring the influence of the sintering temperature (T_s) on the structure of PMT-PNT-PZ-PT ceramic, the best T_s was found to obtain good electrical properties.

In this work, $0.4\text{Pb}(\text{Ni}_{1/3}\text{Ta}_{2/3})\text{O}_3\text{-}0.6\text{PbTiO}_3$ exhibits high dielectric properties. It induces lead vacancies by incorporating Ni^{2+} and Ta^{5+} into the B site of PZT, which facilitates domain switching and effectively enhances the piezoelectric performance of the ceramic, $0.006\text{Pb}(\text{Mn}_{1/3}\text{Ta}_{2/3})\text{O}_3\text{-}0.114\text{Pb}(\text{Ni}_{1/3}\text{Ta}_{2/3})\text{O}_3\text{-}0.43\text{PbZrO}_3\text{-}0.45\text{PbTiO}_3$ (PMT-PNT-PZ-PT) is designed to simultaneously enhance d_{33} and Q_m , showing promising potential for high-power device applications.

2. Materials and Methods

$0.006\text{Pb}(\text{Mn}_{1/3}\text{Ta}_{2/3})\text{O}_3\text{-}0.114\text{Pb}(\text{Ni}_{1/3}\text{Ta}_{2/3})\text{O}_3\text{-}0.43\text{PbZrO}_3\text{-}0.45\text{PbTiO}_3$ (abbreviated as PMT-PNT-PZ-PT) ceramics were fabricated via the conventional solid-state method. The starting materials used were PbO (99.9%), ZrO_2 (99.99%), NiO (99%), Ta_2O_5 (99.99%), and MnO_2 (99%). All starting materials were weighed according to the stoichiometric ratio and then milled in alcohol with zirconia balls for 12 h. The resulting mixture was dried and pre-burned at 750°C for 2.5 h, and the calcined powder was ball-milled for 24 h again. The powder was mixed with 7 wt.% polyvinyl alcohol (PVA) as a binder, which provided cohesion and green strength for shaping. The mixture was then uniaxially pressed into gray disks with a diameter of 13 mm. Subsequently, the prepared disks underwent a debinding process at 550°C to completely remove the PVA. Finally, the disks were embedded in a sacrificial powder with the same composition as the ceramic matrix to prevent reaction and deformation, followed by sintering for 2.5 h at different temperatures ($T_s = 1180, 1200, 1250$, and 1270°C). To facilitate the characterization of electrical properties, the samples were polished and coated with silver to serve as electrodes.

The crystalline phases of the ceramics were identified using XRD (D/max-rB 12kW X-ray diffractometer). The fractured surface micromorphology of the sintered samples was examined by scanning electron microscopy (SEM, SU5000). The temperature dependence of the dielectric constant and loss was measured with an LCR test instrument (Agilent, E4980A, Santa Clara, CA, USA) from room temperature to 450°C at 0.1 kHz, 1 kHz, 10 kHz, 100 kHz, 1 MHz. For piezoelectric properties, the ceramics were poled in a silicone oil bath at 150°C under a DC field of 3 kV/mm for 15 min. Poling at this elevated temperature lowers the coercive field, thereby facilitating domain alignment under the applied electric field. The piezoelectric coefficient d_{33} was measured by a quasi-static meter (ZJ-4A). The ferroelectric hysteresis loops of the ceramic samples were measured using a ferroelectric test system (Premier II, Radiant Tech, Albuquerque, NM, USA). The mechanical quality

factor (Q_m) and electromechanical coupling factor (k_p) were calculated based on IEEE standards using an Agilent 4294A Precision Impedance Analyzer, as follows:

$$Q_m = \frac{1}{4\pi(f_a - f_r)R_1(C_0 + C_1)} \quad (1)$$

$$\frac{1}{k_p^2} = 0.395 \times \frac{f_r}{f_a - f_r} + 0.574 \quad (2)$$

3. Results and Discussion

Figure 1 presents the XRD patterns of PMT-PNT-PZ-PT ceramics sintered at different temperatures (1180 °C, 1200 °C, 1250 °C, and 1270 °C). The positions of the main diffraction peaks remain essentially unchanged with sintering temperature, indicating that the perovskite structure is maintained and no secondary phases appear. Although this observation alone does not directly prove dopant incorporation into the lattice, the subsequent variations in electrical properties (discussed below) are consistent with the expected effects of Mn, Ni, and Ta entering the B-site of the perovskite lattice, as widely documented for similar PZT-based systems. All diffraction peaks were indexed based on the tetragonal perovskite structure (JCPDS No. 70-4260), and no peaks corresponding to secondary phases were observed, indicating the formation of a pure perovskite solid solution. The crystal structure is identified as tetragonal for the PMT-PNT-PZ-PT ceramics, as evidenced by a (200)/(002) peak intensity ratio of approximately 1:2. Furthermore, the (110) diffraction peak shows a subtle variation in intensity with sintering temperature, with the highest intensity observed at 1200 °C. This trend is consistent with literature reports on PZT-based ceramics, where intermediate sintering temperatures promote optimal crystallinity, while excessive temperatures may introduce lattice distortion or lead loss that reduces peak intensity.

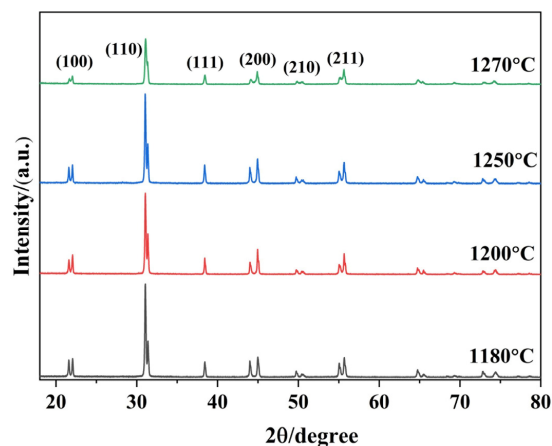


Figure 1. XRD pattern of PMT-PNT-PZ-PT ceramics at different sintering temperatures.

Figure 2a–d displays the SEM micrographs of PMT-PNT-PZ-PT ceramics sintered at different temperatures. The samples sintered at 1180 °C and 1200 °C exhibit a dense microstructure, well-developed grains, and no visible pores or abnormal grain growth, suggesting excellent ceramic quality and optimal electrical properties at these temperatures. The grain size as a function of sintering temperature is summarized in Figure 2e,f. As the sintering temperature increases, the average grain size shows a clear upward trend, indicating that higher temperatures promote grain growth. However, the emergence of pores at elevated temperatures likely reduces the densification of the ceramics, thereby degrading their electrical performance. In the sample sintered at 1270 °C, the grains are closely bonded and exhibit a tendency to coalesce, which can be attributed to the partial melting of over-sintered grains.

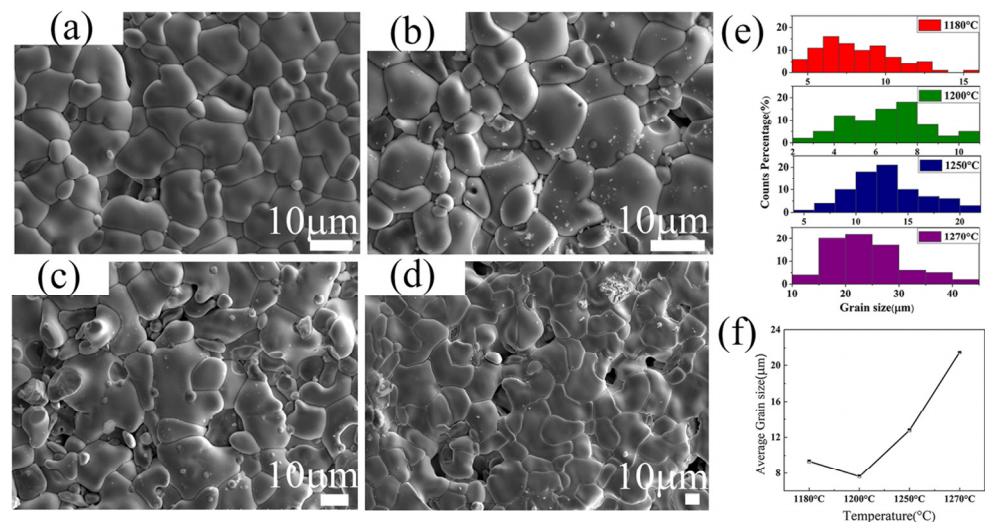


Figure 2. SEM microstructure of calcined ceramic at different sintering temperatures (a) 1180 °C, (b) 1200 °C, (c) 1250 °C, (d) 1270 °C, (e) Grain size distribution, (f) Variations of average grain size.

Figure 3a–d illustrates the temperature dependence of the dielectric constant and loss for PMT-PNT-PZ-PT ceramics measured from room temperature to 450 °C at frequencies of 0.1 kHz, 1 kHz, 10 kHz, 100 kHz, 1 MHz. A single permittivity peak is observed near the Curie temperature (approximately 300 °C) for all compositions, indicating a ferroelectric–paraelectric phase transition. At room temperature, the ceramics exhibit a purely tetragonal phase, which is consistent with the XRD analysis. The decrease in $\tan\delta$ with increasing frequency is characteristic of dipolar relaxation, which in perovskite ferroelectrics is often associated with the presence of oxygen-vacancy-related defect dipoles [33,34]. At the low frequency, $\tan\delta$ start to rapidly increase beyond T_c due to thermally activated space charge conduction behavior [35].

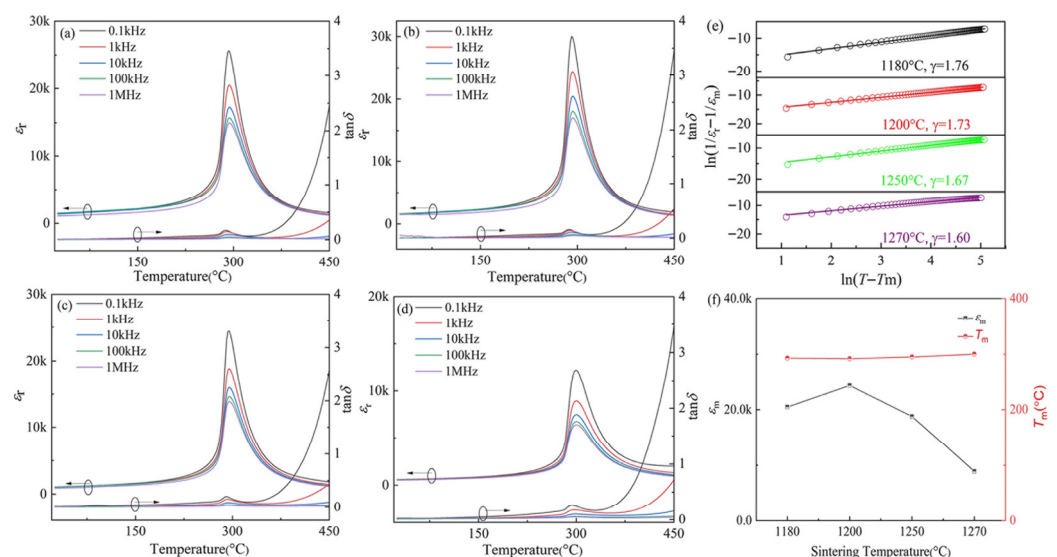


Figure 3. Temperature dependence of dielectric constant and dielectric loss of PMT-PNT-PZ-PT ceramics with different frequencies at different sintering temperatures: (a) 1180 °C, (b) 1200 °C, (c) 1250 °C, (d) 1270 °C, (e) Modified Curie–Weiss fitting curves of PMT-PNT-PZ-PT ceramics sintered at different temperatures, (f) Variations of ϵ_m , T_m .

To further analyze the dielectric behavior, a modified empirical formula was employed to evaluate the dielectric dispersion and diffusivity (γ) of the phase transition. The calculation of γ based on the Curie–Weiss law can be formulated as follows:

$$1/\varepsilon - 1/\varepsilon_m = (T - T_m)^\gamma / C \quad (3)$$

where ε_m is the maximum dielectric constant at T_m . Figure 3e further reveals that all samples exhibit characteristic relaxor ferroelectric behavior with a diffuse phase transition, regardless of sintering temperature. As the sintering temperature increases, the diffuseness parameter γ gradually decreases, suggesting that grain growth tends to suppress the relaxor characteristics. Notably, in the sample sintered at 1180 °C, the dielectric loss ($\tan\delta$) remains below 0.02 at 10 kHz. This low-loss profile is highly favorable for applications such as ultrasonic transducers.

The P - E loops of PMT-PNT-PZ-PT measured at room temperature are shown in Figure 4. With the increase of sintering temperature, P - E loops gradually become asymmetrical. The presence of acceptor–oxygen-vacancy defect dipoles leads to the generation of an internal bias field [36–38]. The internal bias field can be calculated by

$$E_i = \frac{|E_c^+ - |E_c^-||}{2} \quad (4)$$

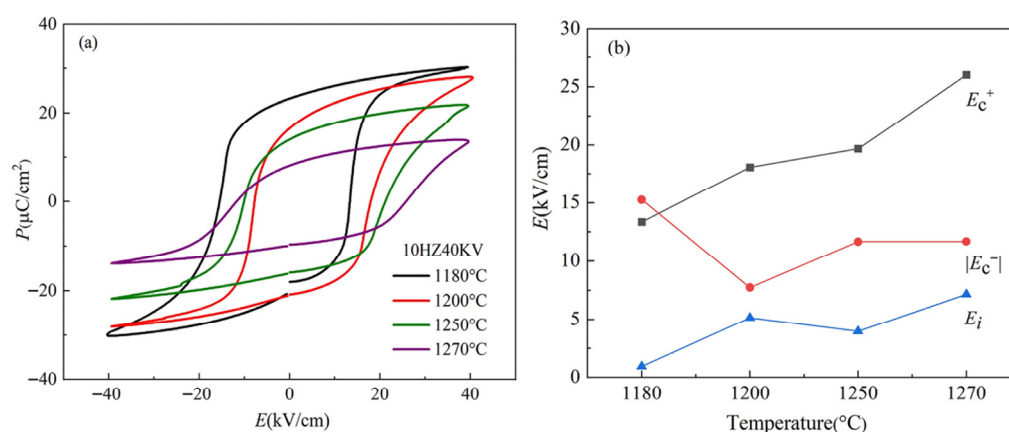


Figure 4. Ferroelectric properties of PMT-PNT-PZ-PT ceramics: (a) P - E hysteresis loops, (b) The coercive field of positive electric field (E_c^+), negative electric field (E_c^-), and the internal bias field (E_i).

Figure 4b shows the changing rule of E_c^+ , $|E_c^-|$ and E_i as a function of sintering temperature. The E_i increases proportionally with the sintering temperature, whereby higher temperatures lead to elevated concentrations of oxygen vacancies and defect dipoles, ultimately resulting in an augmented internal bias field. Here, ‘defect dipole’ refers to the association between an acceptor ion and an oxygen vacancy, which aligns under an electric field and gives rise to an internal bias field. The increase in E_i with sintering temperature suggests a higher density of such defect dipoles, likely due to enhanced oxygen vacancy concentration at elevated temperatures.

Figure 5 describes the relationship between d_{33} , Q_m and sintering temperature. The d_{33} value gradually decreases with the sintering temperature, while the Q_m value initially increases and then subsequently decreases. In order to assess the optimal comprehensive properties of PMT-PNT-PZ-PT ceramics, figure of merit (FOM) is defined as $FOM = d_{33} \times Q_m$ [39,40]. According to Figure 5, the optimum integrated performances of d_{33} (400 pC/N), Q_m (509), and FOM (203,600 pC/N) were found for the sintering temperature of 1180 °C. The observed trends can be explained by the incorporation of Mn ions and the associated generation of oxygen vacancies for charge compensation. These point defects act as pinning centers for domain walls, restricting their motion [41,42]. This pinning effect is responsible for the gradual decrease in d_{33} and the initial increase in Q_m with sintering temperature, as domain wall contributions to dielectric and piezoelectric

responses are reduced while mechanical losses are minimized. For perovskite ferroelectrics, the piezoelectric coefficient d_{33} can be expressed as [43]:

$$d_{33} = 2P_r Q \epsilon_0 \epsilon_r \quad (5)$$

where P_r is residual polarization, ϵ_r is the dielectric constant, ϵ_0 is the dielectric constant in vacuum, and Q is the electrostrictive coefficient (for the same material Q is the same). When the sintering temperature is 1180 °C, the ceramics have the largest piezoelectric constant (d_{33}). Combined with Figure 5b, the variation of d_{33} and $P_r \epsilon_r$ is consistent, so the large d_{33} comes from $P_r \epsilon_r$. The analysis shows that the Mn enters the lattice at higher temperatures. To uphold the electrical neutrality of the cell, a greater number of oxygen vacancies are generated within the material. The existence of oxygen vacancies hinders the mobility of the domain wall, resulting in a decrease in d_{33} and k_p , while Q_m exhibits an increase. Incorporation of Mn ions into the system induces ceramic “strengthening” effects, leading to an enhancement in Q_m .

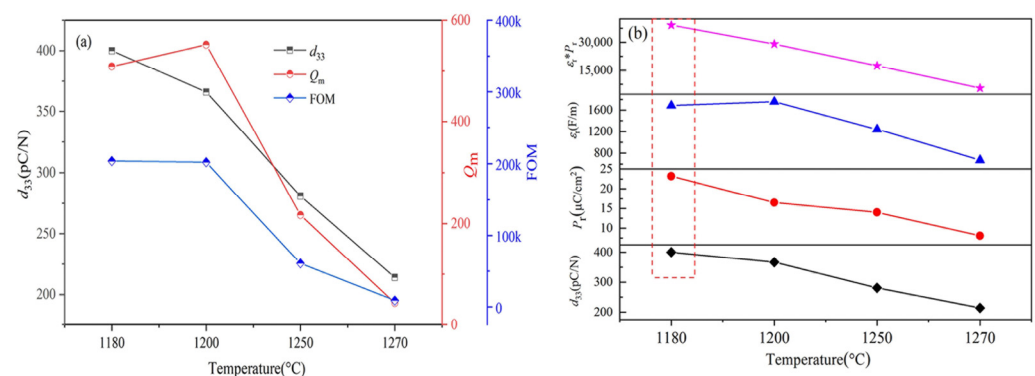


Figure 5. (a) Values of Q_m , d_{33} and FOM with the sintering temperatures, (b) The electrical properties of PMT-PNT-PZ-PT ceramics sintered at different temperatures.

The properties of the ceramics are compared with those of commercial PZT4 ceramics and other ceramics, as presented in Table 1. We effectively reconcile the trade-off between d_{33} and Q_m , thereby presenting a novel approach to enhance the sensitivity of ultrasonic transducers while minimizing heat generation. Manganese ions can exist in multiple valence states (Mn^{2+} , Mn^{3+} , Mn^{4+}). In the PZT lattice, Mn preferentially occupies B-sites (Zr^{4+} , Ti^{4+}). If Mn enters as Mn^{2+} or Mn^{3+} , it acts as an acceptor, creating negative effective charges (e.g., $Mn^{''}Zr$ for Mn^{2+} on Zr^{4+} site, or $Mn^{'}Zr$ for Mn^{3+}). To maintain electroneutrality, oxygen vacancies ($V^{O\bullet\bullet}$) with effective positive charges are formed. These acceptor–oxygen vacancy pairs ($Mn^{''}Zr-V^{O\bullet\bullet}$) constitute defect dipoles that pin domain walls, leading to increased Q_m and reduced d_{33} . The degrees of acceptor incorporation and oxygen vacancy concentration increase with sintering temperature, explaining the observed trends. Furthermore, it reveals significant advantages in various other properties compared to the aforementioned piezoelectric ceramics.

Table 1. Comparison of electrical properties between the present PMT-PNT-PZ-PT ceramic (sintered at 1180 °C) and other representative piezoelectric ceramics.

Material	ϵ_r	k_p	k_t	T_c (°C)	d_{33} (pC/N)	Q_m	FOM(pC/N)	Ref.
PMT-PNT-PZ-PT (1180 °C)	1686	0.65		304	400	509	2.04×10^5	This work
PZT4	1300	0.58		328	289	500	1.50×10^5	[44]
PMN-PZT				216	1530	100	1.53×10^5	[45]
BS- <i>y</i> PT- <i>x</i> BMS	1384	0.50		410	330	84	2.80×10^4	[46]
KNNS-BNZ- <i>x</i> BZ	3460	0.58	0.45		610	34	2.10×10^4	[47]

4. Conclusions

The effects of sintering temperature on the phase structure and electrical properties of $0.006\text{Pb}(\text{Mn}_{1/3}\text{Ta}_{2/3})\text{O}_3\text{-}0.114\text{Pb}(\text{Ni}_{1/3}\text{Ta}_{2/3})\text{O}_3\text{-}0.43\text{PbZrO}_3\text{-}0.45\text{PbTiO}_3$ (PMT-PNT-PZ-PT) ceramics were comprehensively investigated. The sintering temperature plays a critical role in tailoring the microstructure and electrical performance of PMT-PNT-PZ-PT ceramics. The ceramic sintered at 1200 °C exhibited the smallest grain size along with the highest mechanical quality factor (Q_m) and relative permittivity (ϵ_r). The piezoelectric constant d_{33} (400 pC/N) is achieved at 1180 °C, accompanied by a significant $P_r\epsilon_r$ (39,115 μCcm^{-2}). Compared to commercial PZT-4 ceramics, the composition sintered at 1180 °C attained an optimal balance between d_{33} and Q_m , resulting in a superior comprehensive figure of merit ($\text{FOM} = 2.04 \times 10^5 \text{ pC/N}$). These results not only provide a viable candidate material for high-power and temperature-stable piezoelectric devices but also offer valuable insights into the processing–property relationships in complex perovskite ceramics.

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Conflicts of Interest: Authors Junjun Wang and Junjun He were employed by the company Harbin Kaina Technology Co., Ltd., and Liqiang Liu was employed by the company Harbin Hatran Navigation Technology Co., Ltd. The remaining authors declare that the research was conducted in the absence of any commercial or financial relationships that could be construed as a potential conflict of interest.

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