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Effect of $PbZr_{0.53}Ti_{0.47}O_3$ concentration on the degree of phase transition diffuseness and depolarization temperature in transparent ceramics $Pb(Mg_{1/3}Nb_{2/3})O_3 - xPbZr_{0.53}Ti_{0.47}O_3$

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The effect of the PZT component content (x) on the degree of phase transition diffuseness, as well as on the relative position of the depolarization temperature (T_d) and the morphotropic phase transition (T_{F-R}) was studied in transparent ceramics $Pb(Mg_{1/3}Nb_{2/3})O_3{-}xPbZr_{0.53}Ti_{0.47}O_3$ (PMN$-x$PZT)($x = 10$, 16, 23, 33 %). Dielectric, optical and pyroelectric measurements were carried out for this purpose. It was shown that all the studied ceramics are relaxors, and in the compositions with $x = 23$, 33 % a spontaneous first-order ferroelectric phase transition is observed in the absence of an electric field. It was found that in all compositions a single-stage transition of the polarized sample to the relaxor phase occurs, i. e. the temperatures T_d and T_{F-R} coincide. The obtained results are discussed in terms of the degree of phase transition diffuseness and the sizes of the polar regions.

Keywords: transparent ceramics, relaxors, ferroelectricity, induced phase transition.

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1. Introduction

Among all lead-containing complex perovskites the ceramic of lead zirconate-titanate $Pb(Zr,Ti)O_3$ or PZT was studied most widely both from scientific and practical point of view [1–4]. Special attention was paid to the compositions at the morphotropic phase boundary (MPB) with ratio Zr/Ti 52/48 and 53/47 due to the best electric properties in a wide area of applications. Besides, different PZT modifications with the element substituted with donor or acceptor admixtures were also widely covered in the literature [5–7]. Many studies were conducted on PZT modification with a relaxor ferroelectric, lead magno-niobate ($Pb(Mg_{1/3}Nb_{2/3})O_3$ or PMN), since it is well known that the ceramic of solid solution of relaxor and normal ferroelectrics is a rather promising material for electronic devices, such as capacitors, actuators, sensors, converters and instruments for ultrasonic medical diagnostics [8–17].

Indeed, PZT modification with PMN is interesting, since it presents a combination of the desired properties with high dielectric permittivity and high electromechanical coupling coefficient, and has low losses, which is especially important for use in various devices [11,12].

Recently the transparent ferroelectric ceramics has been attracting more and more attention of the researchers due to some advantages compared to transparent single crystals, such as low cost, simple manufacturing, good control of ingredients etc. [18]. It has great prospects of use in the areas of electrooptic switches and modulators, due to a noticeable electrooptic (EO) effect. Transparent ferroelectric ceramic $PbZrTiO_3$, alloyed by La (PLZT), was consistently studied for many years due to high EO effect compared to transparent single crystals $LiNO_3$ [19–22]. However, PLZT ceramics has high losses for scattering depending on polarization, and significant signal delay caused by the field, which limits its use in high-frequency dynamic devices.

Recently developed transparent ceramic PMN$-x$PZT has no such disadvantages. In our papers [23–26] we were first to prepare transparent ceramic PMN$-x$PZT with $x = 10$, 16, 23 with high transparency (~ 65 %) and high value of electrooptic effect. The least transparency was observed in the compositions with $x = 33$ % (~ 40 %), which is due to larger ferroelectric macrodomains, while compositions PMN$-x$PZT ($x = 10$, 16, 23) had small ferroelectric domains.

It was found that all ceramic specimens studied in the paper had a relaxor-like behavior, besides, the more the PZT content was, the less the phase transition was diffused. It was found that in the compositions that were closer to MPB ($x = 23$ and 33 %), in absence of the electric field, a ferroelectric first-order phase transition is observed, while the compositions further away from the boundary ($x = 10$, 16 %), remain in the relaxor cubic phase up to the low temperatures. It was found that in the compositions with $x = 33$ % at high temperatures in paraelectric phase the value of the quadratic EO-coefficient was the highest value for ferroelectrics at such high temperatures.

Papers devoted to these systems don't contain any information about the relationship between the depolarization temperature of pre-polarized samples T_d and the temperature of ferroelectric-relaxor phase transition T_{F-R}. Temperature T_{F-R} is very important for piezoelectric applications, because it is the limit over which materials loose their macroscopic properties. For most of applications, T_d is also an important parameter since piezoelectric and pyroelectric effects depend to a lesser degree on the long-range order between local dipoles and to a greater degree on the capability to maintain macroscopic residual polarization.

As is known from the literature and our papers [27–32], these temperatures may coincide with each other in a number of relaxors such as PLZT, PMN, PST, PSN, and may differ, for example, in relaxors such as PZN, PZN-PT solid solutions, $Na_{1/2}Bi_{1/2}TiO_3$ (NBT), solid solutions of NBT with $BaTiO_3$ (NBT-BT) and $(K_{0.5}Na_{0.5})NbO_3 - 0.02Ba_2NaNb_5O_{15}$ (KNN-BNN).

Coincidence of temperatures T_d and T_{F-R} may occur in ferroelectrics both with high and low degree of phase transition smearing. In case of significant smearing the quantity of polar areas is great, and they have small dimensions. With a decrease in temperature, the PNR sizes increase, the distance between them decreases down to Vogel-Fulcher temperature T_f, below which the PNR sizes remain almost the same, but quite small ($\sim 10-30$ nm), nonergodic glass phase appears. In an electric field the ferroelectric phase is induced in the course of time. When the polarized specimen is heated at temperature T_d, it breaks not only the macroscopic polarization, but, as a result of produced small-size PNR, the interconnection is lost between local dipoles inside the domains, i. e. the process of depolarization and transition of the specimen to the relaxor phase happens practically at the same temperature, i. e. temperatures T_d and T_f coincide.

In relaxors with the minimum degree of phase transition smearing PNR in the high-temperature ergodic phase are ordered units of rather big size, and interactions between PNR cause a spontaneous phase transition order-disorder in ferroelectric state in the absence of the electric field. The number and concentration of polar regions, being insignificant at the Curie point and with lowering of PNR temperature, can increase in size considerably up to macroscopic ferroelectric domains. Temperature of the spontaneous phase transition, which establishes the ferroelectric long-range order, is close to the temperature of the dielectric permittivity maximum. The T_f temperature is nearly the same as the temperature of the clear phase transition and, therefore, the depolarization temperature. Since usually theT_f temperature is described as the temperature of freezing of the system to the frustrated glass state, its use in this case is not necessary.

Mismatch of temperatures T_d and T_f is found only in those relaxors, where a diffused phase transition is observed in absence of electric field into a ferroelectric state. The depolarization process in them takes place in two stages.

At the first stage heating of the polarized sample over T_d destroys the macroscopic polarization only. The domains start vibrating due to thermal activation, but the interrelation between the local dipoles within the domains is not lost. At the second stage, when the material is heated to a temperature of T_{F-R} and above, the domains are decomposed to PNR.

In the PMN$-x$PZT ceramic studied in this paper the dominant component is PMN, where the ferroelectric phase transition takes place only in the presence of the electric field, and temperatures T_d and T_{F-R} coincide [33]. PZT is not a relaxor. In process of work it is suggested to find out how the change in the content of PZT(x) component impacts the mutual location of temperatures T_d and T_{F-R}. Dielectric, optical and pyroelectric properties will be measured with this purpose.

2. Examined samples and experimental procedure

Highly transparent ceramic PMN$-x$PZT ($x = 10$, 16, 23, 33) with the ratio Ti : Zr equal to 53 : 47, was prepared using an unusual two-stage sintering method described in our paper [24]. In order to increase the probability of perovskite phase formation and to decrease the presence of pyrochlore phase in manufacture of the ceramics, lead zirconate-titanate was used instead of lead zirconate. The produced specimens were of high optical quality. Dielectric and optical properties of these transparent ceramics were studied in detail in our papers [23–26] and in paper [34]. A universal E7-11 was used for dielectric measurements. Measurements of dielectric permittivity (ε) and tanδ were carried out at frequency 1 kHz in the temperature range of $290-400$ K in two modes of electric field application: when heated in the absence of the electric field (ZFH) and when heated after specimen cooling in the electric field (ZFHaFC). To study the temperature dependences of birefringence Δn, He-Ne-laser was used. Measurement of thermally stimulated depolarization (TSD) or pyrocurrent is described in detail in our paper [35]. Specimens were cooled in the outer field 3 kV/cm. Polarization was calculated by integrating the TSD current density with time.

3. Experimental results and discussion

Figure 1 (*a–d*) presents temperature dependences of dielectric permittivity ε (curves *1*) and dielectric loss angle tangent tanδ (curves *2*)), obtained in process of heating in absence of the electric field (ZFH mode), and also tanδ (curves *3*), obtained in mode (ZFHaFC) after cooling in the field 3 kV/cm for all studied specimens.

From the figure you can see that as PZT content increases, temperatures of maxima ε and tanδ move towards high temperatures. For all specimens in absence of the electric field only one maximum ε and tanδ is observed, besides for one and the same specimen the temperatures of maxima ε and tanδ do not coincide (curves *1, 2)*, which indicates diffused phase nature of the transition from the relaxor to the paraelectric phase. No other drastic abnormalities, for example, compliant with MPT transition, are observed. A small bend may be observed in temperature dependence ε in the area of 360 K for $x = 33$ % (Figure 1, *d* — curve *1*) and 320 K for $x = 23$ %

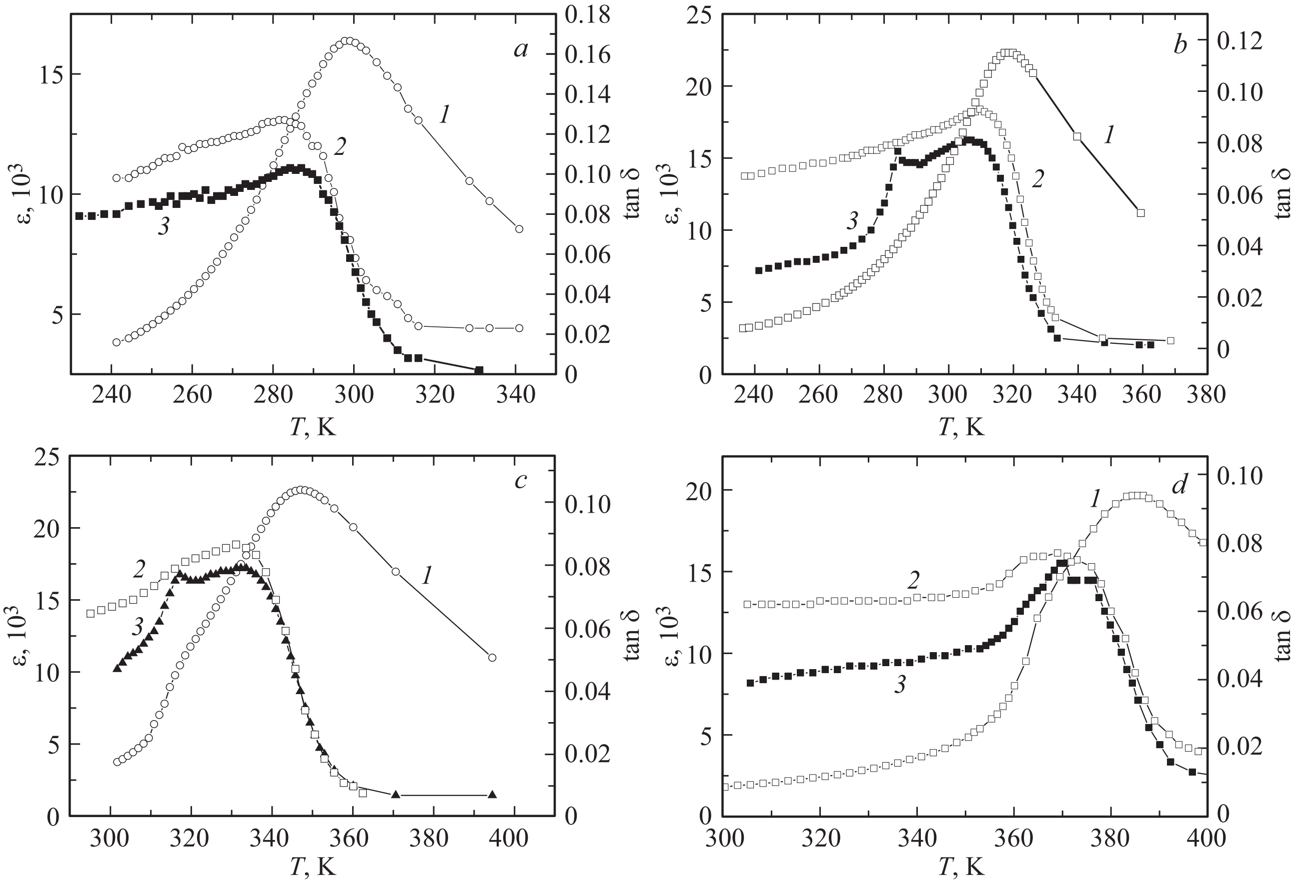

Figure 1. Temperature dependences of dielectric permittivity ε (1) and dielectric losses tanδ (2), obtained in process of heating in absence of the electric field (ZFH mode) (curves *1, 2*) and tanδ (3), obtained in mode (ZFHaFC) after cooling in the field 3 kV/cm for specimens PMN$-x$PZT: *a* — $x = 10\,\%$, *b* — $x = 16\,\%$, *c* — $x = 23\,\%$, *d* — $x = 33\,\%$

(Figure 1, *c* — curve *1*). This bend in curve ε in process of specimen heating may be related to MPT from ferroelectric rhombohedral (monoclinic) phase to relaxor tetragonal phase, which is damaged near the maximum temperature ε. These compositions are close to a normal ferroelectric, since they are located not far from the morphotropic area.

Abnormalities manifest more clearly in the specimens polarized in the electric field. Indeed, clear maximum at MPT temperature appeared in curves tanδ (curves *3*) in specimens with $x = 16$, 23, 33 %. In specimen with $x = 10\,\%$, located far from MPT, at MPT temperature $\mathrm{T}_{F-R} \sim 250-260\,\mathrm{K}$ (Figure 1, *a* — curve *3*) only a minor diffused abnormality is observed.

Figure 2 presents dependences of dielectric permittivity measured at room temperature, and temperature of maximum ε on content of PZT (x). You can see that with increase in content of x temperature $T_{\max\varepsilon}$ moves monotonously towards higher temperatures with average speed of ~ 4 K/mol%. These data agree well with the results of papers [11,16]. Value ε at room temperature decreases with growth of x.

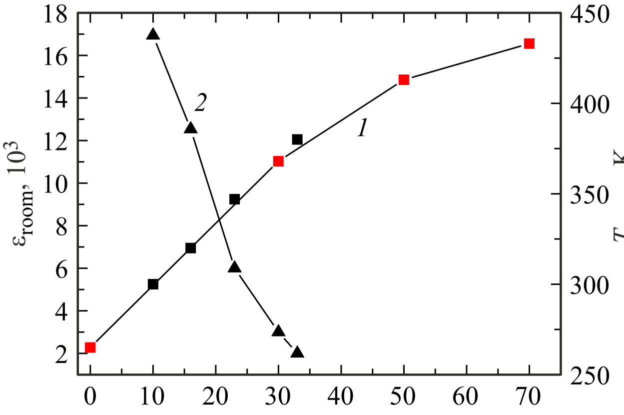

Figure 2. Dependences of maximum temperature ε (curve *1*) and dielectric permittivity measured at room temperature (curve *2*), on content of PZT (x). Data indicated with red dots are taken from paper [16].

You can see the impact of PZT(x) content at the degree of smearing of the phase transition more clearly in Figure 3. Figure 3 presents concentration dependences of the difference between the temperatures of maxima ε and tanδ ($T_{\max\varepsilon} - T_{\max\tan\delta}$) (curve *1*), and also the difference between temperatures of maxima ($T_{\max\varepsilon}$) and morphotropic

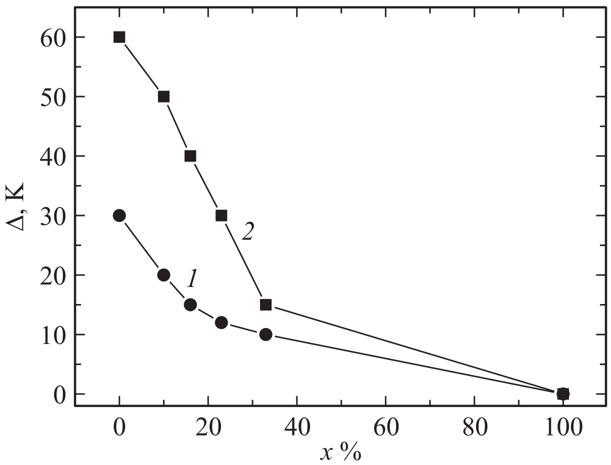

Figure 3. Concentration dependences of the difference between temperatures $(T_{\max\varepsilon} - T_{\max\tan\delta})$ (curve *1*) and $(T_{\max\varepsilon} - T_{F-R})$ (curve *2*) on content of PZT(x).

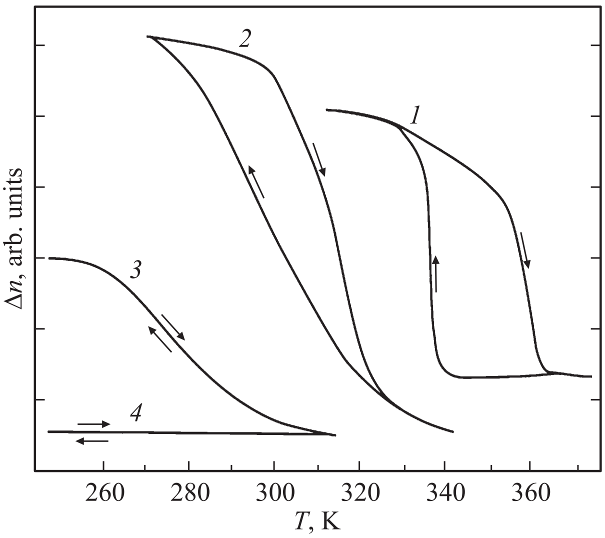

Figure 4. Dependences Δn on temperature obtained in cooling (ZFC) and heating (ZFH) modes in the absence of the electric field for specimens PMN$-x$PZT with $x = 33$ (*1*), 23 (*2*), 16 (*3*), 10% (*4*).

phase transition T_{F-R} $(T_{\max\varepsilon} - T_{F-R})$ (curve *2*) on content of PZT(x).

From Figure you can see (curve *1*), that as content of x increases, temperatures of maxima tanδ and ε approach each other, i.e. the area of relaxor phase existence narrows. The distance between the temperatures of morphotropic phase transition T_{F-R} and $T_{\max\varepsilon}$ (curve *2*) also reduces with increase of x, and compositions with $x > 33\,\%$ practically behave as normal ferroelectrics with very low share of relaxor phase. Symmetry of compounds with x content higher that 33% is purely tetragonal, and at temperature of $T_{\max\varepsilon}$ the transition from the tetragonal phase to the cubic phase occurs omitting the relaxor phase. Such compositions have all matching specific temperatures, such as $T_{\max\varepsilon}$ and T_{F-R}, according to [36]. As concentration of PZT(x) increases, the gradual transition occurs from the relaxor to the normal ferroelectric state. In the ceramic samples studied herein, the normal ferroelectric state was not achieved.

Figure 4 presents temperature dependences of birefringence Δn, obtained in the mode of cooling (ZFC) and heating (ZFH) in absence of the electric field for all compositions studied in the paper.

Optical research methods are more sensitive compared to, for example, dielectric methods, especially for studying the changes of sizes of inhomogeneities in case of phase transitions. This is confirmed by data of Figure 4. Thus, in compositions with $x = 23$ and 33% in process of cooling from high temperatures at MPT temperature, birefringence appears (curves *1*, *2*), while in the temperature dependence ε the abnormality at MPT is practically invisible (Figure 1, *c*, *d*, curve *1*). Birefringence is related to increase in the size of heterogeneities exceeding the light wavelength. The observed temperature hysteresis ~ 10 K for composition $x = 23\,\%$ and ~ 20 K for composition $x = 33\,\%$ indicates a spontaneous first-order phase transition in these compositions.

For composition with $x = 16\,\%$ (Figure 4, curve *3*) in process of cooling, only increase Δn is observed, being related to gradual increase in the number and dimensions of ferroelectric areas in the cubic relaxor matrix. No phase transition occurs into macrodomain state in this composition, since there is no hysteresis in cooling and heating. In composition with $x = 10\,\%$ Δn will not occur down to low temperatures (Figure 3, curve *4*), i.e. dimensions of ferroelectric areas are too small, and the specimen remains in the cubic relaxor state.

As you can see from the above figures, no additional abnormalities, for example, at temperature of depolarization T_d, when dielectric and optic properties are measured, were found in the studied ceramics. Then you may conclude that probably temperatures T_d and T_{F-R} coincide.

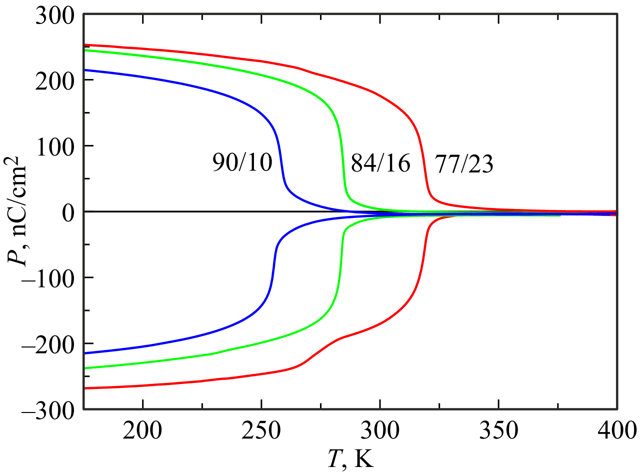

Figure 5. Temperature dependences of residual polarization obtained by integration of thermally stimulated depolarization current.

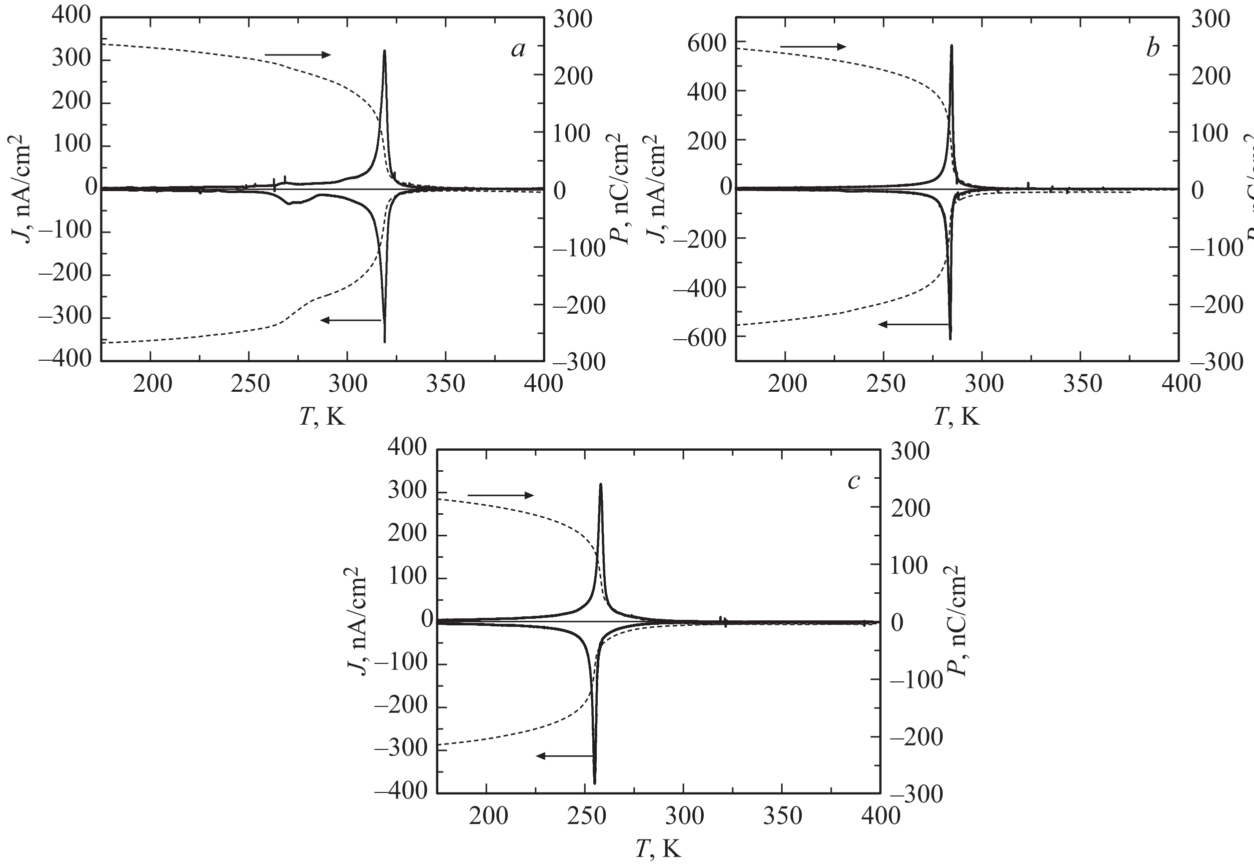

Figure 6. Temperature dependences of pyroelectric current and residual polarization for ceramics $x = 23\,\%$ (*a*), $x = 16\,\%$ (*b*) and $x = 10\,\%$ (*c*).

To verify this assumption, we measured pyrocurrent and residual polarization. Temperature T_d — is the temperature of depolarization of a previously polarized specimen. T_{F-R} is the temperature of transition from ferroelectric to relaxor phase. T_d — is the temperature, at which the maximum of pyroelectric current is observed, which corresponds to the bend point in the curve of residual polarization.

Figure 5 presents temperature dependences of residual polarization for ceramics $x = 23\,\%$, $x = 16\,\%$ and $x = 10\,\%$. You can see from the figure that depolarization of all three specimens happens drastically, and the polarization value increases with the increase of x.

Figure 6, *a*, *b*, *c* presents temperature dependences of pyroelectric current (current of thermally stimulated depolarization) for ceramics $x = 23\,\%$ (*a*), $x = 16\,\%$ (*b*) and $x = 10\,\%$ (*c*). The dotted curve shows temperature dependence of residual polarization.

From the Figure you can see that at the same temperature, at which the bend occurs in the curve of residual polarization, both maximum of pyrocurrent and maximum of tanδ (Figure 1 curves *3*), corresponding to temperature T_{F-R} are observed. This transition temperature is the temperature of disintegration of frozen polar configuration from macrodomain state to microdomain one.

From Figure 6 you can see that in all specimens, despite the different structure of low-temperature phase, temperatures T_d and T_{F-R} coincide. Relaxors may have two different states at low temperatures: non-ergodic or ergodic relaxor state. In one relaxors cooling from high temperature to below temperature $T_f < T_{\max\varepsilon}$, non-ergodic relaxor phase occurs, and in the other ones — ergodic relaxor phase [37]. When the electric field is applied, non-ergodic relaxor state irreversibly transforms into ferroelectric state, which when heated is damaged at temperature above T_{F-R} (T_d), which is very close to T_f, and ergodic relaxor state occurs. Ergodic relaxor state observed at low temperatures reversibly changes to ferroelectric phase in the absence of the field damaged when heated at temperature of spontaneous transition close to T_{F-R} (T_d). Indeed, in ceramics with $x = 10$, $16\,\%$, as it follows from Figure 4, in the absence of the electric field there is no spontaneous phase transition, and low-temperature phase is non-ergodic relaxor phase. In this case, when the polarized specimen is heated, due to the formed PNR of small size, at temperature T_d not only macroscopic polarization disappears, but also the interrelation of local dipoles inside domains, i. e. temperatures T_d and T_{F-R} coincide.

In a relaxor with $x = 23\,\%$, according to optical measurements (Figure 4) there is a spontaneous phase transition in the absence of the electric field, and low temperature phase is ergodic ferroelectric phase. Ferroelectric interactions between PNRs result in a ferroelectric macroscopic order.

Temperature T_f is nearly the same as the temperature of the clear phase transition and, therefore, the depolarization temperature T_d.

It should be noted that pyrocurrent peak width is very narrow in all studied ceramics. In paper [12] the authors studied the temperature dependence of resisual polarization and pyrocurrent in ceramic PMN−0.11PZT. Temperatures of maxima of pyrocurrent from our measurements and from paper [12] coincide, however, the half-width of peaks in our paper is only several degrees, whereas in paper [12] — more than 50 degrees.

One of the possible reasons for wide maximum of pyrocurrent in [12] may be a mixed phase structure and co-existence of polar areas of different symmetry and size. Applied electric field induced partial monodomainization of the sample and ferroelectric long-range order only in a part of the sample volume and, consequently, to the co-existence of ferroelectric rhombohedral (or monoclinic) phase domains and tetragonal relaxor phase domains in a wide temperature range. These domains with different symmetry are arranged differently in external fields and have different transition temperatures. Narrow peak of depolarization current that we found in all studied specimens indicates that reduction to a single-domain state occurred in the entire volume simultaneously, and the applied electric field is sufficient for reorientation of all domains in the field. Most likely, this means a practically homogeneous structure of low-temperature phase. The presence of such narrow peak indicates good quality of studied ceramics. In our paper [24] when we studied optical transmission in PMN−xPZT ($x = 10$, 16 %) ceramics, we found that they have very high optical transparency ~ 65 % for the visible and infrared areas of the spectrum. In general, obtaining highly transparent ceramic is a very complicated problem, including many factors, such as powder synthesis, sintering process, density, phase structure, composition homogeneity, properties of grain boundaries and many other factors. In the same paper in process of studies on a scanning electron microscope we found that specimens PMN−xPZT ($x = 10$, 16 %) show a completely tight microstructure without evident observed pairs and thin irregular ferroelectric domains, which provides for high transparency and homogeneity of the composition. Note that from the data given in [24], in PMN−xPZT ($x = 23$, 33 %) ceramic the domains are bigger than in ceramics with $x = 10$, 16 %, which resulted in reduction of optical transmission in the visible area of the spectrum to PMN−23PZT ceramic down to 50 %, and in PMN−33PZT - down to ~ 40 %.

4. Conclusion

This paper studied the impact of PZT (x) component in transparent $Pb(Mg_{1/3}Nb_{2/3})O_3 - xPbZr_{0.53}Ti_{0.47}O_3$ (PMN−xPZT) ($x = 10$, 16, 23, 33 %) ceramic at the degree of smearing of phase transition, and also at the mutual location of depolarization temperature (T_d) and morphotropic phase transition (T_{F-R}). It is shown that as x increases, the ceramic behavior changes: compositions with low value of $x = 10, 16$ % manifest purely relaxor properties, and when cooled from high temperatures below temperature $T_f < T_{\max\varepsilon}$ a nonergodic relaxor phase arises therein.

Compositions with $x = 23$, 33 %, apart from relaxor behavior, undergo a spontaneous phase transition of 1 order to ferroelectric phase in the absence of the electric field, and low-temperature phase is an ergodic phase. It was found that in all specimens, despite different structure of low-temperature phase, at one and the same temperature, corresponding to the temperature of the morphotropic phase transition T_{F-R}, maximum of pyrocurrent (thermally stimulated depolarization current), bend in the curve of residual polarization and maximum tanδ of the polarized specimen are observed. This indicates a single-stage transition of the polarized specimen to the relaxtor phase, i.e. temperatures of depolarization T_d and T_{F-R} coincide. Narrow peak of depolarization current and drastic change of residual polarization that we found in all studied specimens indicates that reduction to a single-domain state occurred in the entire volume simultaneously, and the applied electric field is sufficient for reorientation of all domains.

The assumption was made that the presence of such narrow peak indicated good quality of the studied ceramics and practically homogeneous structure of the low-temperature phase.

Conflict of interest

The authors declare no conflict of interest.

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