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Influence of the Degree of Phase Transition Diffuseness on the Depolarization Temperature in Relaxors of Different Types

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The relationship between the temperatures of depolarization (T_d) and morphotropic phase transition ($T_{\mathrm{F-R}}$) in crystalline relaxor solid solutions of various types, such as $\mathrm{PbMg_{1/3}Nb_{2/3}O_3{-}29PbTiO_3}$ (PMN−29PT), $\mathrm{PbZn_{1/3}Nb_{2/3}O_3{-}9PbTiO_3}$ (PZN−9PT) and $\mathrm{NaBi_{1/2}Ti_{1/2}O_3}-x\mathrm{BaTiO_3}$ ($x = 5, 7.5\%$) (NBT−xBT) has been studied. For this purpose, dielectric measurements of polarized samples were carried out, and the process of induction of the ferroelectric phase in an electric field applied below the $T_{\mathrm{F-R}}$ temperature was also studied. It was found that the structure of the low-temperature phases in these compounds is different, which leads to significant differences not only in the induction of the ferroelectric phase, but also to different relative positions of the temperatures T_d and $T_{\mathrm{F-R}}$. In PMN−29PT, the formation of ferroelectric phases is preceded by some delay time, which is one of the hallmarks of a non-ergodic glassy phase, and in this case the temperatures T_d and $T_{\mathrm{F-R}}$ coincide. In PZN−9PT and NBT−5BT, the ferroelectric phase is induced immediately after the field is applied without a delay time, which indicates that below the $T_{\mathrm{F-R}}$ temperature, the non-ergodic glassy phase does not appear, and the temperatures T_d and $T_{\mathrm{F-R}}$ do not coincide in them. The results obtained are discussed from the point of view of different degrees of diffuseness of the phase transition and different sizes of the polar regions. It is suggested that the coincidence of temperatures T_d and $T_{\mathrm{F-R}}$ is a consequence of the non-ergodic glassy phase and the small sizes of the polar regions.

Keywords: relaxors, phase transitions, degree of smearing, depolarization temperature.

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1. Introduction

For the last decades, relaxor ferroelectrics attract increased attention of researchers due to their superior dielectric and piezoelectric properties, that are widely used in numerous devices. These properties are associated with the presence of polar nanoregions (PNR). At high temperatures (several hundred degrees higher than the temperature corresponding to the maximum dielectric permittivity), relaxors are in the nonpolar paraelectric cubic phase, which is similar in many respects to the paraelectric phase of normal ferroelectrics. As the temperature decreases, relaxors undergo a transition to the ergodic relaxor state wherein PNRs (2−10 nm), which give rise to the peculiar features of relaxors, emerge at the Burns temperature $T_{\mathrm{B}} \sim 620{-}650\,\mathrm{K}$.

There are several characteristic temperatures in relaxors below the temperature T_{B}. The temperature for the maximum dielectric permittivity ($T_{\max}$), which is dependent on frequency and approaches the freezing temperature (T_f — Vogel–Fulcher temperature) at zero frequency, if there is no spontaneous ferroelectric transition of sample in this type of relaxors. By definition, temperature T_f is the temperature of the static dielectric permittivity maximum at zero frequency, $T_{\max}$. This temperature, T_f, can be estimated from frequency-dependent dielectric permittivity data recorded while cooling the sample at a constant cooling rate by a numerical fit using the Vogel–Fulcher relationship in analogy to glass transition. However, this method is quite labor-intensive because it requires a wider range of measurement frequencies than the frequencies from 100 Hz to 1 MHz commonly used for experiments. In addition, the Vogel–Fulcher equation has three unknown parameters to be fitted. Therefore, instead of T_f, $T_{\mathrm{F-R}}$ or T_d temperatures are used that are by far easier to determine from experiments and sufficient for most practical applications. Temperature $T_{\mathrm{F-R}}$ is the temperature of transition from ferroelectric to relaxor phase. Temperature T_d is the temperature of depolarization of a previously polarized sample. Temperature $T_{\mathrm{F-R}}$ is very important for piezoelectric applications, because it is the limit over which materials loose their macroscopic properties [1]. In [2,3] it was shown that the depolarization process observed at T_d has a common origin with the temperature $T_{\mathrm{F-R}}$. Indeed, in some relaxors, such as 9/65/35PLZT and $\mathrm{PbMg_{1/3}Nb_{2/3}O_3}$ (PMN), it was found that freezing temperature coincides with depolarization temperature [4]. However, this coincidence of temperatures should be considered carefully, because they often are different from each other. This is the case, first of all, of the relaxors where depolarization occurs in a wide range of temperatures, as it takes place, for example, in materials based on sodium-bismuth titanate. In [5–7], as well as in our study [8], it was shown that depolarization temperature T_d is by 30−40° lower than

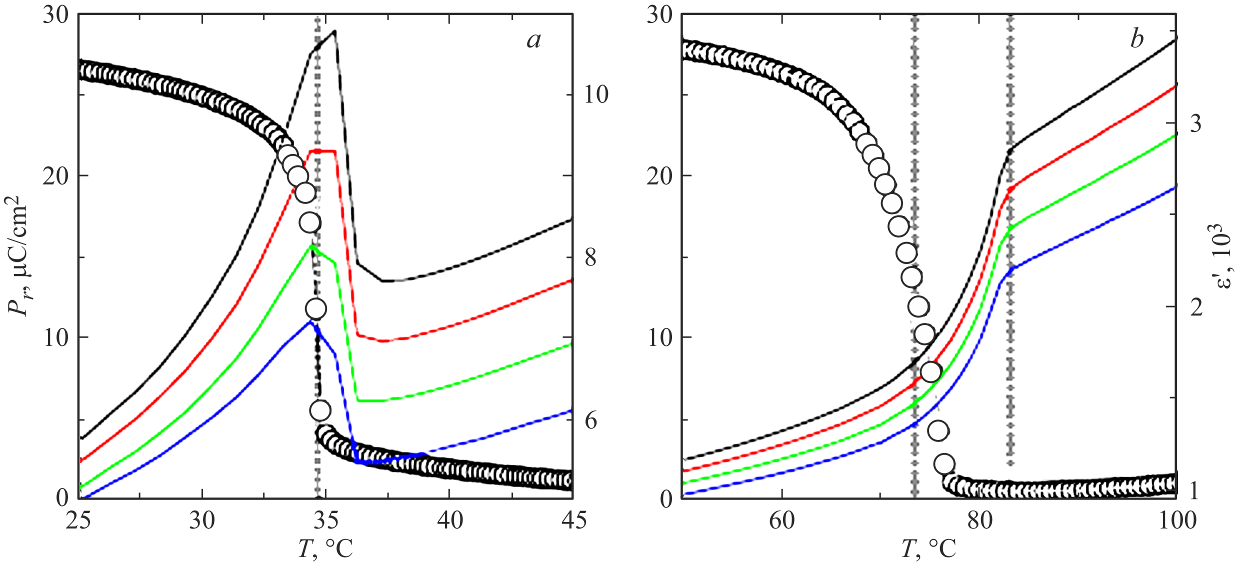

Figure 1. Comparison of temperatures T_d and T_{F-R} for PLZT 8/65/35 (*a*) and NBT−6BT (*b*) measured from the temperature dependence of dielectric permittivity obtained at frequencies of 0.1, 1, 10,100 kHz and the remanent polarization derived from thermally induced depolarization current measurements [9].

temperature T_{F-R}. T_d is the temperature where maximum piezoelectric current is observed, which corresponds to the tipping point on the remanent polarization curve. T_{F-R} coincides with the initial temperature of inverse dielectric permittivity deviation from linear behavior, i.e., with the beginning of a significant growth of dielectric permitivity. Fig. 1 shows as an example the comparison of depolarization temperature T_d and T_{F-R} for PLZT 8/65/35 Fig. 1, *a* and solid solution $Na_{1/2}Bi_{1/2}TiO_3-6BaTiO_3$ (NBT−6BT) Fig. 1, *b* from [9].

It can be seen from the figure that depolarization of PLZT 8/65/35 sample takes place abruptly at the temperature where maximum dielectric permittivity is observed. Conversely, the rate of remanent polarization change in NBT−6BT compounds (Fig. 1, *b*) is much lower, that results in the significant deviation of temperature T_d from the dielectric anomaly corresponding to T_{F-R}, which is matched with literature data [1,2].

There is no single point of view regarding the observed difference of T_d and T_{F-R} temperatures in some relaxors. Thus, the authors of [6] associate the difference in temperatures in NBT−6BT solid solutions with the fact that depolarization in them is a two-stage process.

At the first stage heating of the polarized sample over T_d destroys the macroscopic polarization only. The domains start to vibrate due to thermal activation, but the interrelation of the local dipoles inside 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.

The authors of [10], studying ceramic samples of $Pb_{0.99}[Zr_{0.45}Ti_{0.47}(Ni_{0.33}Sb_{0.67})_{0.08}]O_3$, explain the loss of polarization at temperatures T_d lower than the long-range order break at T_{F-R}, by the existence of depolarizing fields and stresses caused by the non-compliance of deformation at grain boundaries due to different crystal-lattice orientations of grains and distortion of the lattice structure at the transition.

These explanations leave unclear, why in some relaxors the coincidence of T_d and T_{F-R} temperatures is observed, and in others there is no such coincidence. In this study we made an attempt to present our point of view regarding this non-compliance. For this purpose, dielectric properties of polarized samples will be studied, as well as the process of inducing the ferroelectric phase and the kinetics of nucleation of ordered ferroelectric phases in an electric field applied below the T_{F-R} temperature in relaxors of different types. We shall analyze the obtained results and their interrelation with other experimental results published in literature.

2. Examined samples and experimental procedure

We have selected 2 classes of crystalline relaxors as the research target. The first class includes PMN solid solutions with $PbTiO_3$ (PT) (for example, PMN−29PT). The dominant component in PMN−29PT is PMN where a ferroelectric phase transition takes place only in an electric field. From literature data it is known [4], that T_d and T_f temperatures coincide with each other in PMN.

The second class of relaxors includes solid solutions of sodium-bismuth titanate $NaBi_{1/2}Ti_{1/2}O_3$ (NBT) with $BaTiO_3$ (BT) NBT−5BT and NBT−7.5BT, as well as solid solutions of $PbZn_{1/3}Nb_{2/3}O_3$ (PZN) with $PbTiO_3$ (PT) (PZN−9PT). In these compounds there is a diffused ferroelectric phase transition in the dominant component NBT and PZN in the absence of the electric field. In [5–7], as well as in our study [8], it was shown that in both pure NBT and NBT$-x$BT with low x T_d and $T_{\mathrm{F-R}}$ temperatures do not coincide with each other.

In these compounds the dependencies of dielectric permittivity vs time will be measured for annealed samples in an electric field. The samples were cooled down without a field to a temperature below the temperature of a morphotropic phase transition, at which a constant electric field was applied to them. Following the application of the electric field, the process of variation of the permittivity was timed and recorded. In NBT$-$5BT and NBT$-$7.5BT compounds dependencies of dielectric permittivity (ε) and $\tan\delta$ vs temperature were measured for previously polarized samples.

In all studied samples without the electric field applied the macroscopic cubic phase is kept down to low temperatures, and they all demonstrate a relaxor behavior.

3. Experimental results and discussion

3.1. PMN and PMN$-$29PT

The structure of classical relaxor (PMN) remains cubic with nanometric inclusions of polar regions down to low temperatures. The ferroelectric phase may emerge in it either in an electric field or when the $(\mathrm{Mg}_{1/3}\mathrm{Nb}_{2/3})$ complex is substituted partially with Ti^{4+} ions. It is known fairly well how the ferroelectric phase transition is induced in PMN, and PMN$-x$PT solid solutions [11–16], as well as in some other relaxors. In the process of cooling from the ergodic paraelectric phase, PNRs (2$-$10 nm in case of PMN) grow in size down to temperature T_f. At temperatures lower than T_f, the size of PNRs remains almost constant (2$-$10 nm for PMN), and only the orientation of their dipole moments changes. A nonergodic glass phase with frozen local polarization emerges, and the ferroelectric phase transition in PMN is induced only in an electric field. The wide spectrum of relaxation times and the presence of the incubation period during the induction of the ferroelectric transition with a long-range order in the electric field is one of the proofs in favor of nonergodicity of the relaxor (glass) phase.

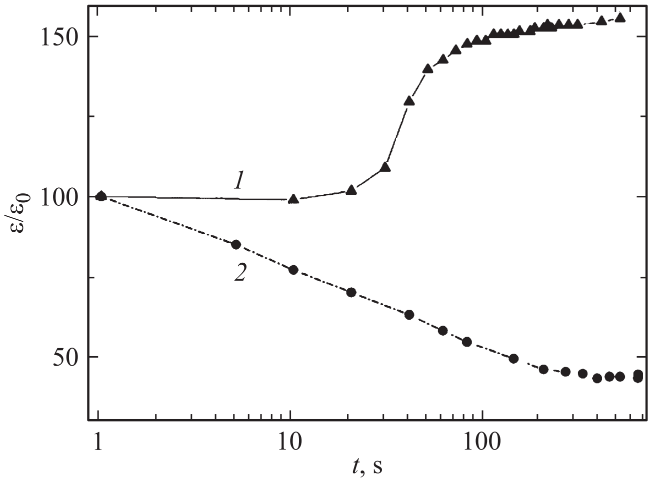

Figure 2. Time dependences of the dielectric permittivity measured at room temperature for PMN$-$29PT crystals (curve *1*) in an electric field of 1.23 kV/cm and PZN$-$9PT (curve *2*) in an electric field of 3.3 kV/cm (ε_0 is the dielectric permittivity in the zero field).

Fig. 2 shows time dependences of the dielectric permittivity measured by us at room temperature for PMN$-$29PT crystals (curve *1*) in an electric field of 1.23 kV/cm and PZN$-$9PT (curve *2*) in an electric field of 3.3 kV/cm ε_0 is the dielectric permittivity in the zero field). It can be seen that in PMN$-$29PT a certain time delay, which is indicative of a nonergodic glass phase, precedes the formation of ferroelectric phase and the rapid establishment of macroscopic polarization in an electric field applied at temperatures below the MPT temperature ($T_{\mathrm{F-R}} \sim 360-370$ K). According to models of structural glass [17,18], the nonergodic glassy state manifests as a result of freezing of thermal-activated dipole moments of nanoclusters, which, as known, exist in relaxors. The sizes of rhombohedric PNRs remain unchanged below the MPT temperature; the only thing that changes is the orientation of their dipole moments.

Previously in [17,19–21] the authors, studying PMN$-x$PT ($0 < x < 35$) solid solutions, have found the coincidence of the Vogel–Fulcher temperature (of the MPT temperature for high concentrations x) with the depolarization temperature T_d derived from the dependence of remanent polarization vs temperature.

It can be assumed that the coincidence of T_d and $T_{\mathrm{F-R}}$ temperatures is a consequence of the nonergodic glass phase and small size of polar regions in the PMN (10$-$30 nm) and solid solutions based on it. The number of polar regions is high due to large diffusing of the phase transition. With a decrease in the PMN temperature, the PNR sizes increase, the distance between them decreases down to the T_f temperature, below which the PNR sizes remain almost the same, but quite small ($\sim$ 10$-$30 nm). In an electric field the ferroelectric phase is induced in the course of time. When a polarized sample is heated, at a temperature of T_d not only macroscopic polarization is destroyed, but also, due to the emerging small-sized PNRs, the interrelation of local dipoles inside domains is lost. It means that in PMN the process of polarization and transition of the sample to the relaxor phase takes place at nearly the same temperature, i.e. T_d and T_f coincide with each other. A similar situation is observed in PMN$-$29PT solid solution as well.

3.2. PZN and PZN$-$9PT, NBT and NBT$-x$BT ($x = 5, 7.5$)

As can be seen from literature review and our report [8], non-coincidence of T_d and $T_{\mathrm{F-R}}$ temperatures is observed in some compounds with a quite large difference between them. These compounds include NBT and NBT$-x$BT [22,23]. Fig. 3 shows as an example the

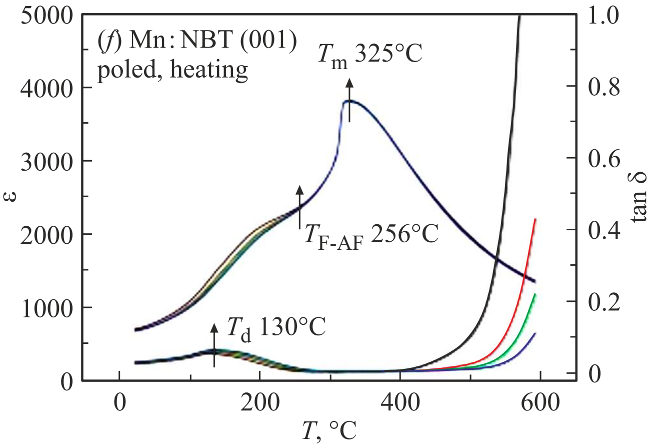

Figure 3. Dielectric permittivity and $\tan\delta$ as a function of temperature measured while heating a polarized sample of NBT + Mn from [22].

temperature dependencies of dielectric permittivity (ε) and $\tan\delta$ for a polarized sample of NBT + Mn, taken from [22]. It can be clearly seen from the figure, that in NBT T_d and $T_{\mathrm{F-R}}$ temperatures are different from each other.

Fig. 4, *a, b* shows temperature dependencies of ε and $\tan\delta$ measured by us for NBT−5BT (*a*) and NBT−7.5BT (*b*) crystals.

It can be clearly seen from Figs. 3 and 4, *a, b*, that in both pure NBT crystal and in solid solutions of NBT−5BT and NBT−7.5BT T_d and $T_{\mathrm{F-R}}$ temperatures are different from each other. The depolarization temperature for a polarized crystal of NBT−5BT (Fig. 4, *a*) corresponds to a little maximum of $\tan\delta$, while at the temperature of $T_{\mathrm{F-R}}$ a tipping point is observed on the temperature dependence of ε.

In a polarized sample of NBT−7.5BT (Fig. 4, *b*), the peak of ε at ~ 388 K corresponds to a depolarization temperature of the sample. $T_{\mathrm{F-R}}$ temperature at 465 K corresponds to a change in the slope of the ε curve in a non-polarized sample. These results match the literature data [23–25].

Our studies of the dependencies of dielectric permittivity vs time in an electric field for PZN−9PT and NBT−5BT crystals measured at a temperature below the temperature of the morphotropic phase transition are shown in Fig. 2 (curve *2*) and Fig. 5 [8,26].

It can be seen from these figures that the ferroelectric phase is induced in these compounds immediately after the application of an electric field without any delay, which is indicative of the fact that the nonergodic glass phase most likely does not emerge.

The abrupt increase in ε immediately after the electric field is switched on and the emergence of the maximum in Fig. 5 may be associated with the activation of processes of tetragonal domains switch-over, which are observed in the fields greater than the coercive field, as well as with an increase in the mobility of domain walls in the process of sample polarization. The ongoing polarization may include the formation of macroscopic tetragonal domains. After the maximum an abrupt decrease in $\varepsilon/\varepsilon_0$ is observed, which is indicative of the beginning of transition into the ferroelectric phase (supposedly, the tetragonal one) with the macroscopic domain structure.

As far back as in the early Tagantsev study [27], it was shown that the Vogel–Fulcher relationship for the dependence of dielectric permittivity maximum vs frequency used in relaxors can be derived as a direct consequence of gradual spectrum broadening with a decrease in temperature, and does not necessarily assume „freezing“ of PNR in such system.

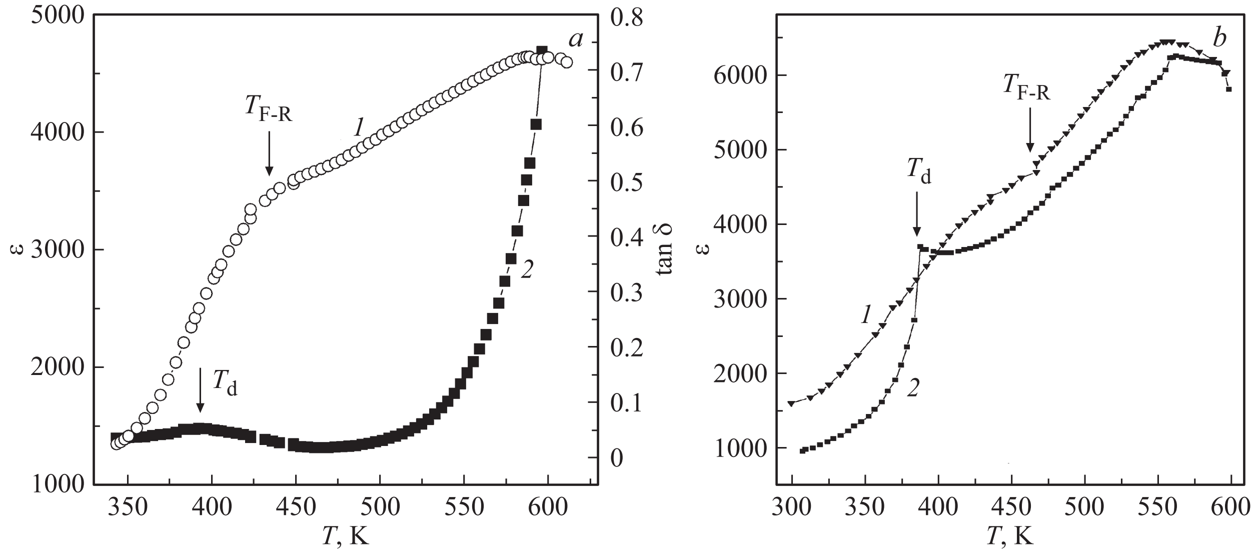

Figure 4. Temperature dependencies of ε (curve *1*) and $\tan\delta$ (curve *2*) of a polarized sample of NBT−5BT (*a*) and non-polarized (curve *1*) and polarized sample (curve *2*) of NBT−7.5BT (*b*) measured while heating.

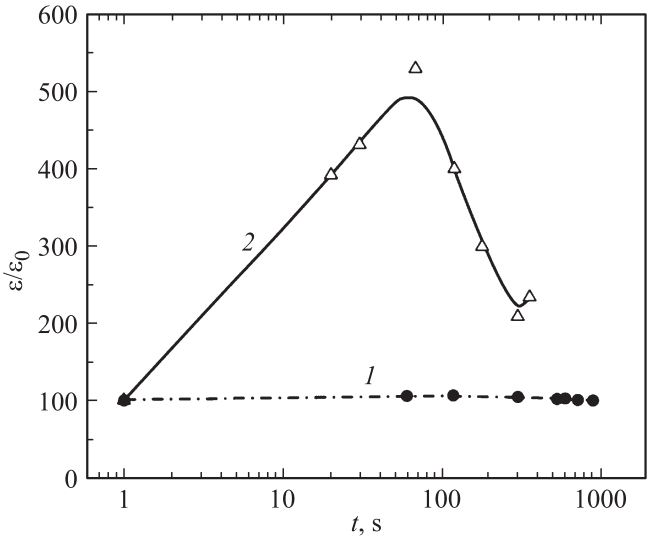

Figure 5. Time dependences of $\varepsilon/\varepsilon_)$ at a room temperature for NBT$-$5BT crystals in an electric field of 10 kV/cm (curve *1*) and 13.8 kV/cm (curve *2*), where ε — dielectric permittivity in the presence of the field at various moments of time, ε_0 — dielectric permittivity in the zero field.

Our results shown in Fig. 2 (curve *2*) and Fig. 5 have confirmed the conclusions of [27] regarding the unnecessary freezing of PNR sizes below the Vogel–Fulcher temperature. The absence of delay time may be connected with the continuing growth of polar region sizes even below the Vogel–Fulcher temperature. It was suggested in [28,29] that the ferroelectric phase in PZN is more likely to be induced by the polar nanoregion (PNR) sizes increase than by the interaction between PNRs, since it is this interaction that initiates the emergence of the glass-like nonergodic relaxor state.

Indeed, in the crystals under study, in a dominant PZN and NBT component, without the electric field there is a diffused phase transition into the ferroelectric state, in contrast to PMN [29,30]. It means that the blurriness degree of the phase transition is less than for the PMN and, therefore, the PNR concentration is less, while distances between them are larger. The higher temperature of the phase transition in PZN, as well as in NBT, as compared with PMN, probably promotes to the formation of larger polar regions, since at high temperatures the possibility occurs to reorient the dipole moments of some PNRs due to thermal motion. The sizes of rhombohedral PNRs in these compounds are considerably larger than those in PMN. Thus, in PZN they are $\sim 40-200$ nm, while in NBT they are ~ 100 nm [29]. With a decrease in temperature PNRs have a space for size increase up to mesoscopic domains, however, less than the size of normal ferroelectric domains. The growth of PNRs into ferroelectric domains takes place at different temperatures for different PNRs, that is they grow gradually in a wide range of temperatures, so the dielectric permittivity changes without sharp anomalies.

It can be assumed that non-coincidence of T_d and $T_{\mathrm{F-R}}$ temperatures is only observed in compounds where there is a blurred phase transition to the ferroelectric state without an electric field, and this is associated with the sizes of polar regions. For example, when PZN crystals are cooled from the paraelectric phase, domains of the ferroelectric phase emerge at a temperature of $T_{\mathrm{C}} \approx 390$ K, then they grow very quickly due to the decrease in the volume of the cubic phase, and below ≈ 325 K most of the sample is transferred to the ferroelectric phase, however in general the structure remains cubic. Whereas the beginning and the end of transition do not coincide with each other, a polarized sample with heating transits to the relaxor phase gradually as well: first the macroscopic polarization is lost at $\mathbf{T}_d$, and then, at T_{C}, final transition to the relaxor phase takes place. A similar pattern can be also observed in NBT. Thus, it should be noted once again, that T_d temperature does not necessarily switch on the total loss of the polarization state of the material.

PZN occupies an intermediate position between another two classes of relaxors, such as PMN and disordered crystals of $\mathrm{PbSc_{1/2}Ta_{1/2}O_3}$ (PST) and $\mathrm{PbSc_{1/2}Nb_{1/2}O_3}$ (PSN). In disordered crystals of PST and PSN without an electric field a spontaneous order–disorder phase transition to the ferroelectric state is observed, which is accompanied with a sharp peak on the dielectric permittivity curve. In these disordered compounds PNRs in the high-temperature ergodic phase are ordered units. Ferroelectric interactions between PNRs result in a ferroelectric macroscopic order. T_{C} temperature of this transition is close to the temperature of maximum dielectric permittivity. The parameter of phase transition blurring in these compounds is the lowest among all relaxors. As a result of this 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. After the T_{C} temperature, the ferroelectric long-range order is established.

In [31,32] it was shown that in PST and PSN compositionally disordered crystals the Vogel–Fulcher temperature T_f is nearly the same as the temperature of maximum of the temperature dependence of static susceptibility (T_{A} is close to T_{max}) and the temperature of the spontaneous phase transition T_{C} and, therefore, the temperature of depolarization. Usually, the T_f temperature is described as the temperature of freezing of the system to the frustrated glass state. On the basis of this a conclusion has been made that T_f in PST and PSN does not necessarily mean the temperature of freezing into nonergodic glass state and its use in this case is not necessary.

It can be concluded from the above results, that non-coincidence of $\mathbf{T}_d$ and T_f temperatures is only observed in reflexors where there is a blurred phase transition to the ferroelectric state without an electric field. In PZN$-x$PT, NBT$-x$BT solid solutions and in a number of other relaxor compounds, sizes of polar regions increase and the portion of the cubic phase decreases with an increase in the contents

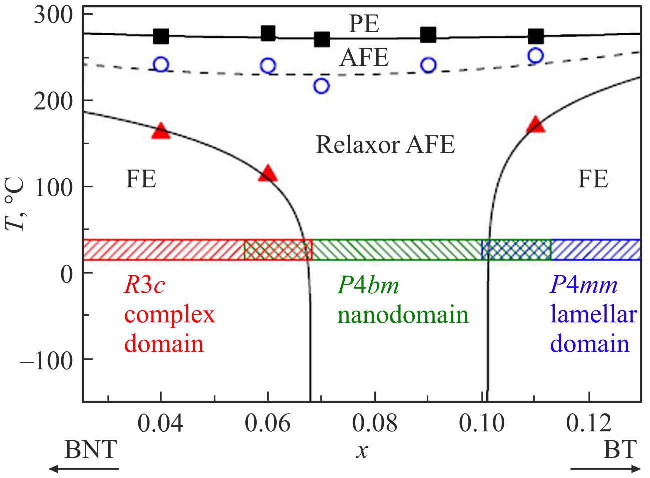

Figure 6. Phase diagram for NBT−xBT non-polarized ceramics. Square dots, circles, and triangles determined from dielectric measurements correspond to T_m, $T_{\mathrm{F-R}}$, and T_d [25] temperatures.

of the second component (increase in x). The diffused phase transition to ferroelectric state observed in them at low values of x in the absence of an electric field is gradually transformed to a clear phase transition. $\mathbf{T}_d$ and T_f temperatures should become closer to each other. These conclusions are confirmed by results of [25,33]. Fig. 6 shows a phase diagram for NBT−xBT non-polarized ceramics from [25]. With high content of x ($x > 0.10$), when the ceramics is nearly in the tetragonal phase already, the $\mathbf{T}_d$ temperature approaches the $T_{\mathrm{F-R}}$ temperature.

4. Conclusions

From the analysis of our dielectric measurements and measured dependencies of dielectric permittivity vs time for PMN−29PT, PZN−9PT, and NBT−xBT ($x = 5, 7.5$) crystal compounds, as well as the literature data, the following conclusions can be made regarding positions of depolarization temperatures T_d and the Vogel–Fulcher temperature T_f (or the temperature of the morphotropic phase transition $T_{\mathrm{F-R}}$).

1. T_d and T_f temperatures coincide with each other in relaxors with the highest degree of phase transition blurriness, where the phase transition to the ferroelectric state is only observed in the electric field (PMN and PMN−xPT with low content of x). In these relaxors nonergodic glassy state emerges at temperatures below T_f (or $T_{\mathrm{F-R}}$), which is a result of freezing of thermal-activated dipole moments of nanoclusters. A certain time delay, which is indicative of a nonergodic glass phase, precedes the formation of ferroelectric phase and the rapid establishment of macroscopic polarization in an electric field. An assumption is made that the coincidence of T_d and $T_{\mathrm{F-R}}$ temperatures is a consequence of the nonergodic glass phase and small size of polar regions (in PMN $\sim 10-30$ nm). When a polarized sample is heated, at a temperature of T_d not only macroscopic polarization is destroyed, but also, due to the emerging small-sized PNRs, the interrelation of local dipoles inside domains is lost. It means that the process of polarization and transition of the sample to the relaxor phase takes place at nearly the same temperature, i.e., T_d and $T_{\mathrm{F-R}}$ temperatures coincide with each other.

2. Also, T_d and T_f temperatures coincide with each other in relaxors with the minimum degree of phase transition blurriness (for example, in disordered crystals of PST and PSN), where without an electric field a spontaneous order–disorder phase transition to the ferroelectric state is observed, which is accompanied with a sharp peak on the dielectric permittivity curve. In these disordered compounds PNRs in high-temperature ergodic phase are ordered units and ferroelectric interactions between PNRs result in a ferroelectric macroscopic order. The T_f temperature is nearly the same as the temperature of the clear phase transition and, therefore, the depolarization temperature. Since usually the T_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.

3. The non-coincidence of T_d and T_f temperatures is only found in relaxors (PZN, NBT, and solid solutions based on them, PZN−xPT and NBT−xBT with low x), where the blurred phase transition to the ferroelectric state is observed without an electric field. This is associated with a lower degree of phase transition blurriness as compared with PMN, and, hence, with larger sizes, and lower concentration of PNRs, and larger distance between them (in PZN they are $\sim 40-200$ nm, and in NBT they are ~ 100 nm). The growth of PNRs into ferroelectric domains takes place at different temperatures for different PNRs, that is they grow gradually in a wide range of temperatures, so the dielectric permittivity changes without sharp anomalies. Whereas the beginning and the end of transition do not coincide with each other, a polarized sample with heating transits to the relaxor phase gradually as well: first the macroscopic polarization is lost at $\mathbf{T}_d$, and then, at $T_{\mathrm{F-R}}$, final transition to the relaxor phase takes place. A similar pattern can be also observed in NBT. With temperature decrease even below T_f, the polar regions have space for the size increase up to mesoscopic values, and the ferroelectric phase in them is more likely to be induced by the polar nanoregion (PNR) sizes increase than by the interaction between PNRs, since it is this interaction that initiates the emergence of the glass-like nonergodic relaxor state. The absence of delay time when inducing the ferroelectric phase found by us is indicative of the fact that the phase below T_f is not an ergodic one. A suggestion is made that the absence of the glass-like nonergodic relaxor state results in the non-coincidence of T_d and T_f temperatures.

Conflict of interest

The author declares that she has no conflict of interest.

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