Nontrivial dependence of dielectric stiffness and SHG on \( dc \) bias in relaxors and dipole glasses

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Dielectric permittivity and Second Harmonic Generation (SHG) studies in the field-cooled mode show a linear dependence of dielectric stiffness (inverse dielectric permittivity) on \( dc \) bias in PMN-PT crystals and SHG intensity in KTaO\(_3\):Li at small Li concentrations. We explain this unusual result in the framework of a theory of transverse, hydrodynamic-type, instability of local polarization.

I. INTRODUCTION

Locally disordered ferroelectrics, such as \((1-x)\text{PbMg}_{1/3}\text{Nb}_{2/3}\text{O}_3 - x\text{PbTiO}_3\) (PMN-PT\(^3\)), KTaO\(_3\):Li etc., exhibit unusual properties, which find wide applications\(^4\) For example, PMN-PT crystals from the morphotropic phase boundary (MPB) compositional range possess an extremely high piezoelectric coefficient, which is important to transform electric energy to mechanical and back\(^5\).

The high dielectric permittivity, \( \varepsilon \), and its nonlinearity, i.e. strong voltage dependence of permittivity, makes disordered ferroelectrics very attractive for applications in electrically tunable devices, especially in thin-film form. In these and many other applications (e.g. electrooptic and electrostriction), disordered ferroelectrics are subjects to high levels of \( dc \) biases (\( E \)). In the framework of Landau theory, \( \varepsilon(\varepsilon) \) in cubic crystals is quadratic, at small fields, and only even terms are allowed\(^6\).

It was shown\(^6\) that the giant piezoelectric response in BaTiO\(_3\) and PMN-NT is due to “polarization rotation”, which means that the electric field turns the direction of polarization and produces mechanical stresses. It is important that the potential relief in PMN-PT compositions near the MPB is nearly flat, which allows large strains at comparatively small fields.

These materials have some degree of local disorder that is known to produce polar regions of finite sizes. The rotation of dipole moments in these regions as well as sideways domain wall movements (or the domain wall movements in 90° domains) can contribute to dielectric permittivity in a special way (which we will discuss below), especially, when the field-cooled (FC) procedure is employed (this helps overcoming barriers within the measuring time).

Another model example considering local dipoles is KTaO\(_3\):Li (KLT). Li in KLT is a dipole centre with a large Li-related dipole moment\(^7\) Electric field aligns these dipoles and creates polarization in the direction of the field\(^8\). If one changes the direction of the field in the FC mode then the direction of polarization also changes. That is, the polarization direction follows the direction of the field that should be if the Gibbs statistics works. Each of the Li dipoles has some potential barrier, which should be overcome that produces viscosity. In ferroelectric phases of PMN-PT, some viscosity can arise also because of domain wall movements, as well as because of random fields. The application of the FC procedure allows overcoming this viscosity.

The ability of an idealized dipole (polarization) to rotate (without having anisotropy) implies that this dipole has a transverse instability. Indeed, the application of small fields in the transverse direction results in a huge (ideally infinite) response\(^9\). Random fields, anisotropy, and barriers suppress this effect at small external fields but some features of the transverse instability can be seen at larger fields\(^10\).

In this paper, we present experiments on locally disordered ferroelectrics, PMN-PT. We will show that dielectric stiffness (inverse dielectric permittivity), in ferroelectric phases of PMN-PT, behaves pseudolinearly with the field, above a certain threshold. This behavior does not follow the Landau theory for cubic crystals\(^11\). The suggested in the present paper theoretical description of this effect takes into account third-power contributions to the free energy of ferroelectric phases. The microscopic origin of these contributions is discussed in detail for a model solid solution, KLT, and we explain experimental data\(^12\) on the pseudolinear field dependence of the SHG intensities in KLT at small Li concentrations.

II. EXPERIMENT AND RESULTS

The PMN-PT single crystal samples used in this study were transparent plates cut from a flux grown crystals prepared at the Physics Research Institute of the Rostov State University\(^13\). Large faces of the samples were perpendicular to [100] direction. The sample with composition PMN-PT35 having dimensions of about \( 2 \times 2 \times 1 \) mm\(^3\) was optically polished and annealed at 823 K for half an hour in air, in order to minimize residual stresses.
Platinum electrodes were sputtered on the large faces. Thin (0.05 mm) Pt wires were attached to the electrodes by silver paste to connect the sample with the contacts of a Linkam HFS91: TS 600 hot-stage used as a sample holder. Dielectric measurements were performed at the 2 K/min heating/cooling rate using a computer-controlled impedance analyzer Solartron SI 1260. A blocking circuit protected the impedance analyzer for experiments under high dc bias. Some details of dielectric studies of other PMN-PT crystals have been described elsewhere. Though we focus mainly on the results obtained in the zero field cooling (ZF) and field cooling (FC) modes, field heating (FH), and zero field heating after field cooling protocols were used as well.

The temperature dependences of the complex dielectric permittivity $\varepsilon(T)$ measured in the ZF mode for both PMN and PMN-PT30 single crystals have one diffused frequency-dependent maximum at about 260 K and 400 K (at 1 kHz), respectively (see for details Refs. [13,14]). In order to characterize the PMN-PT35 sample, we show the $\varepsilon(T)$ curves measured in the ZF mode at different frequencies (Fig.1). Two anomalies on the $\varepsilon(T)$ curve are in good agreement with those found in Refs. [13-15,17,18]: a diffused maximum corresponding to the cubic-to-tetragonal phase transition and an inflexion at a lower temperature, $T_1$, corresponding to the MPB (the transition between the tetragonal and rhombohedral (or monoclinic, according to different data [13,15,16,17]) phases. The temperature $T_m$ of the $\varepsilon(T)$ maximum (435 K at 1 KHz) agrees well with the data obtained for PMN-PT crystals of similar compositions. The temperature $T_1$ (=365-375 K at 1 KHz) is comparable with the data for flux-grown crystals with similar $T_m$ values but is substantially (60-100 K) higher than $T_1$ values, reported for PMN-PT crystals with similar $T_m$ values grown by Bridgeman method. Such discrepancy seems to be caused by differences in crystal’s preparation technique and its origin needs additional studies.

In ZF mode, a pronounced dielectric dispersion is observed below $T_m$ in PMN-PT35 and $T_m$ increases with the frequency $f$ of the measuring electric field. The $T_m(f)$ dependence obeys the Vogel-Fulcher relation. While the value of the attempt frequency $f_0 = 5 \times 10^{11}$ Hz is typical for relaxors, the Vogel-Fulcher temperature $T_0 = 430$ K is only slightly lower than $T_m$. Though dielectric dispersion decreases below $T_0$, it remains considerable in the ZF mode (Fig.1). In the FC mode, the dispersion in the tetragonal phase diminishes but, at low $E$’s, remains practically unchanged, close to $T_m$. Such behavior is similar to that observed in disordered PST and PSN-based ceramics and crystals [19,20,21] and seems to be due to the presence of the spontaneous (thermally driven) transition from relaxor to a mixed (ferroelectric/relaxor) phase containing both the nanoscale polar regions and macroscopic ferroelectric domains of tetragonal symmetry. Both the $T_m$ and $T_1$ ex-
hibit thermal hysteresis, in agreement with first order type reported for these phase transitions.\textsuperscript{16,17,18} The degree of the $\varepsilon(T)$ maximum diffusion evaluated using approach\textsuperscript{22} is about 20 K, in good agreement with the data for PMN-PT31 crystals and PMN-PT35 ceramics.\textsuperscript{22}

In the FC mode, the application of even a relatively small dc bias leads to substantial changes of $\varepsilon(T)$. Permittivity in the tetragonal phase decreases with the bias while, in the rhombohedral phase, it increases initially, but, at higher biases, begins to decrease (Fig. 1). As a result, the inflexion transforms into a maximum at $T_1 \approx 363$ K, in good agreement with the data for the [001]-oriented PMN-PT crystals from the 0.31 $< x <$ 0.35 compositional range.\textsuperscript{17,18}

As the dc bias increases, the main $\varepsilon(T)$ maximum, at first, becomes sharper and higher but then lowers, diffuses and shifts to higher temperatures (Fig. 1). These changes are in good agreement with the data for the [001]-oriented PMN-PT crystals from 0.31 $< x <$ 0.35 compositional range as well as with the data on disordered PbSc\textsubscript{1/2}Ta\textsubscript{1/2}O\textsubscript{3} ceramics.\textsuperscript{23} The frequency dispersion of $\varepsilon$ and $T_m$ reduces dramatically for biases exceeding (0.7-1) kV/cm (Fig. 1). Thus, at low $E$, the PMN-PT35 crystal exhibits a relaxor-like behavior, while, at high $E$’s, its properties are similar to ordinary ferroelectrics. The details of the dependence of $T_m$ and $T_1$ on $E$ will be a subject of a separate publication; here we focus on the field dependences of dielectric stiffness.

The main new result, which we want to discuss in the present paper, is the pseudolinear field dependence of dielectric stiffness in the ferroelectric phases of PMN-PT35 above a certain threshold (Fig. 2). Such behavior is not unique. For comparison, Fig. 3 presents dielectric stiffness $\varepsilon$ for [100] PMN-PT30 and PMN crystals. It is seen that, the pseudolinear dependence is fulfilled for these compositions as well, but above a larger threshold.

III. THEORY

A. Hydrodynamic model

At first, we want to discuss experimental data obtained for model compounds, KTaO\textsubscript{3}:Li. This case has been studied carefully on the basis of first-principles computations (see and references therein) and experiment. It was obtained that Li’s are off-center and have a large dipole moment. Li-related dipoles (further, Li-dipoles) can occupy 6 possible positions with equal energy. Hence, each dipole can rotate (FC protocol is assumed). Let a dipole occupy a position along $z$. The application of field $\delta E$ perpendicular to $z$ rotates the dipole. The change of polarization can be expressed as:

$$\delta P_\perp = \chi_\perp \delta E = n \mu \delta E / E$$  \hspace{1cm} (1)

where $n$ is the Li concentration, $\mu$ the dipole moment, $E$ the magnitude of the field along axis $z$, and $\chi_\perp$ the transverse susceptibility: $\chi_\perp$ diverges in this simplified approach but taking into account the random fields and anisotropy of the potential relief stabilizes the system.\textsuperscript{11}

The change of the polarization magnitude in the field can be described by the Landau theory based on the soft mode concept, which we will discuss below. The free energy necessary for the description of the transverse fluctuations has the form

$$\delta F = \frac{1}{2} \int \left[ \chi^{-1}_\perp \delta P_\perp^2 + c (\nabla P_\perp)^2 \right] dV$$  \hspace{1cm} (2)

where $c$ is constant. One can obtain the longitudinal susceptibility from relations between the transverse and longitudinal changes of polarization:

$$\chi_\parallel = \frac{\delta P_\parallel}{\delta E} = \frac{1}{2P} \frac{d}{dE} \left( \delta P_\perp \right)^2 = \frac{n k_B T}{8 \pi \left( \mu c \right)^{3/2} E^{1/2}}$$  \hspace{1cm} (3)

$E > 0$

Notice that this susceptibility is due to transverse fluctuations of polarization. Normally, it should be added to

![FIG. 3: Dielectric stiffness vs the dc bias for [100] PMN-PT30 and [100] PMN single crystals measured at different temperatures under FC protocol at 1 kHz: 350 K (1), 360 K (2), 370 K (3), 200 K (4), 250 K (5). Solid lines are guides to the eye.](image-url)
FIG. 4: The field dependence of the FC SHG intensity in KLT. Solid lines are guides to the eye.

the Langevin susceptibility that will be discussed below. The integral of this equation gives

\[ (P - P_0)^2 = aE \]

\[ P > P_0 \]

\[ E > 0 \]  

where \( a = (n \kappa B T)^2 / 16 \pi^2 \langle \mu \rangle c^3 \) and \( P_0 \) is the FC polarization magnitude at small \( E \) (remnant polarization). Random fields modify this equation of state. It was obtained that equation (4) is true for the field magnitude, which is larger than the random-field magnitude. It is important to say that equation (4) corresponds to a cubic contribution to free energy:

\[ \Delta F = A (P - P_0)^3 - EP \]

\[ P > P_0 \]  

where \( A = a/3 \).

Notice, that the same conclusions can be got under assumption of domain wall contributions to dielectric permittivity. Experimentally, for dielectrics, some violations of Landau expansion over the even terms (valid for the paraelectric phase only) were discussed for PLZT by J-L. Delis; the author connected this with domain wall contributions. The evidence of equation (4) in dielectrics was found in experiments on SHG (in magnetic systems it was discussed in 10,25).

The SHG intensity, \( S \), is proportional to the average square of polarization:

\[ S \sim \left( P_0 + \sqrt{aE} \right)^2 \]

\[ E > 0 \]  

It gives the linear dependence of the FC SHG intensity on \( E \) at \( aE \gg P_0^2 \). This finding is in agreement with the SHG experiment performed for KLT at Li concentration 1.6% (KLT 1.6%), in the FC mode (see Fig. 4).

B. Pseudolinear field dependence of dielectric stiffness

The above consideration is true if only the lattice (soft mode) contribution to polarization is comparatively negligible. Experiments on KLT showed that the lattice (soft mode) contribution to SHG intensity starts at much larger fields than those used in experiment. In some other cases, the contribution of the soft mode may be comparable with the contribution of local dipoles, polar regions or domain walls. We think that this is the case in PMN-PT. Due to low symmetry of ferroelectric phases (we are talking about the phases obtained in the FC mode at \( E \to 0 \)), free energy includes an odd term:

\[ F = \frac{1}{2} \alpha P^2 + \frac{1}{3} A P^3 + \frac{1}{4} b P^4 - EP + ... \]

\[ P > P_0 \]  

The equilibrium condition gives

\[ \alpha P + A P^2 + b P^3 + ... - E = 0 \]

\[ P > P_0 \]  

(8)

Now one can obtain that

\[ \chi^-1 = \alpha + 2AP + 3bP^2 + ... \]

\[ P > P_0 \]  

(9)

At small fields, \( P = -\alpha/A + |\alpha|^{-1}E \) that leads to the observed linear field dependence of inverse susceptibility:

\[ \chi^-1 = -\alpha + 3bP_0^2 + 2A + 6bP_0 \]

\[ |\alpha|^{-1}E + ... \]

\[ E > 0 \]  

(10)

At large fields, one needs to add even terms, and the dependence becomes quadratic and, then, \( E^{2/3} \) as it should.
be in the Landau theory developed for cubic crystals. The interval of the fields, where is true, depends on the coefficients in expansion (10). One can expect this effect close to the phase transition temperature, and at large , which is possible when the transverse spatial fluctuations of polarization are large. Both conditions are satisfied at MPB.

C. Influence of random fields on the FC dependence of dielectric stiffness in PMN

The influence of random fields on hydrodynamic fluctuations of polarization results in the insensitivity of dielectric permittivity to external fields at small fields. Here we will discuss another aspect of this problem. Experiment shows that, in the ferroelectric phases of PMN-PT35, dielectric stiffness pseudolinearly depends on the field in a wide interval of the field. Only at very small fields, where, perhaps, the time of measuring is not enough, there are some deviations from the pseudolinear dependence (Fig. 2). Experimental results obtained for pure PMN show a more pronounced influence of the random fields on dielectric stiffness. It was found that, in the [111] direction, dielectric stiffness of PMN has a bump while, in the [001] direction, dielectric stiffness decreases with the field smoothly. We suggest considering this difference as a result of anisotropy of free energy. We take into account that there is a diffuse first order phase transition in the [111] field.

We consider the free energy in [111] direction corresponding to the first-order phase transition between the cubic and rhombohedral phases:

\[ F(e, T_c) = \frac{1}{2} \alpha P^2 - \frac{1}{4} \beta P^4 + \frac{1}{6} \gamma P^6 - (E + e) P \]  

where \( \alpha = \alpha(T - T_c) \) and \( e \) is random field. From the equilibrium condition, one gets:

\[ \alpha P - \beta P^3 + \gamma P^5 = E + e \]  

Dielectric permittivity can be found by differentiating (12):

\[ \varepsilon'(e, T_c, E) = 1 + \frac{1}{\varepsilon_0} \frac{dP}{dE} = 1 + \frac{1}{\varepsilon_0} \frac{1}{\alpha - 3\beta P^2 + 3\gamma P^4} \]  

In the case \( e = 0 \), polarization experiences a jump at some field \( E_0 \), and dielectric permittivity diverges at this field. Due to the random fields and distribution of \( T_c \)'s, this jump diffuse. This can be described by introducing a distribution function \( f(e, T_c) \) (see the way how to determine the distribution function from experiment in Ref. 30):

\[ \varepsilon_{av}(E) = \int \varepsilon(e, T_c, E)f(e, T_c) \, dedT_c \]  

In order to simplify this complex situation, one can model inhomogeneous matter by introducing a Gauss type distribution of \( E_0 \):

\[ P(E) = P_1(E) + P_2(E) \int \theta(E_0 - E) e^{-(E_0 - E_1)/x_0^2} \, dE_0 \]  

where \( E_1 \) is the position of the dielectric permittivity maximum, \( \theta(x) \) is a step function, \( x_0 \) is the width of the distribution function, \( P_1(E) \) and \( P_2(E) \) are continuous functions. Dielectric permittivity can be got by differentiating (14):

\[ \varepsilon(E) = \alpha_1(E) - \alpha_2(E) \text{erf}[(E - E_1)/x_0] + P_2(E) e^{-(E-E_1)/x_0^2} \]  

here \( \text{erf}(x) \) is the probability integral, \( \alpha_1 \) and \( \alpha_2 \) can be expressed over the derivatives of \( P_1(E) \) and \( P_2(E) \).

Fig. 5 shows the result of the fitting of our expression to experiment. The distribution function width was found at 1.97 kV/cm, and \( E_c = 4.27 \) kV/cm. For the sake of simplicity, we took \( \alpha_1 \) and \( \alpha_2 \) as constants, and \( P_2 \) was proportional to \( E \). The fit shows that the nature of the bump in the dependence of dielectric permittivity on the [111] field can be the diffuse anomaly due to the first order phase transition between the cubic and rhombohedral phases.
IV. DISCUSSION

The performed experiment reveals that dielectric stiffness in the ferroelectric phases of PMN-PT from the MPB range behaves linearly with $E$. By integrating \[ \text{(10)} \] we obtained that the dependence of polarization on $E$ is logarithmic. This dependence includes both even and odd terms although, in the paraelectric phase only even terms are expected. In ferroelectric phases, the odd terms are allowed due to extrinsic contributions. Notice that the paraelectric phase can be a result of ZF procedure. Cooling the same sample at finite $E$ can result in the appearance of remnant polarization. Our experiments have shown that the pseudolinear field dependence of dielectric stiffness is well observed at temperatures near the MPB (where an intermediate monoclinic phase can appear stimulating the polarization rotation), but, in the paraelectric phase, the data are not that definite. Thus the important feature for the observation of the linearization of dielectric stiffness $\epsilon$ vs $E$ is a flat enough potential relief, which is indeed the case in PMN-PT compositions from the MPB range. Note, that in the PMN-PT30 crystal, which is outside of the morphotropic phase boundary region and especially in pure PMN, the pseudolinear portion of the $1/\epsilon (E)$ dependence is observed at larger fields than in PMN-PT35. This is consistent with theory which showed that the onset of the unusual power law in the hydrodynamic model is at the fields, which are larger then the random field magnitude. In that study only the cubic term was considered in the free energy. In the present study we have added the normal quadratic term responsible for the polarization of lattice (soft-mode contribution). We have shown that these two terms together result in the pseudolinear field-dependence of nonlinear permittivity.

Our finding correlates with the recent observation of the linear field dependence of dielectric stiffness in thin films of PMN where the energy barriers between metastable states of polar clusters appear smaller than in bulk and ceramics. Approximately linear dependences of dielectric stiffness on $dc$ bias were observed in disordered PST ceramics near the border of stability of relaxor ferroelectric phase.

At small $P_0$, the expression obtained for dilute KLT provides the uncommon equation of state, $P^2 \sim E$, which resembles that obtained for some magnets. This behavior has been evidenced in experiments on SHG in KTaO$_3$-Li for the Li concentrations 1.6% (Fig. 4), which, in our opinion, reflects the fact of strong transversal spatial fluctuations of polarization. If the ZF procedure were employed then one would not expect such an effect at low $T$ due to the nonergodicity in this system arising because of potential barriers (viscosity). In such experiments only the ordinary, Langevin type, response of the dipoles should be seen. However, if, due to different reasons, a tilt of the Li dipoles is prevented by internal random electric fields or stresses then the fluctuations become weak and the corresponding contribution to the susceptibility vanishes. For example, for the concentrations larger than 2%, the SHG intensity behaves with the field exponentially, $\sim \exp [(\mu E - w)/k_B T]$ (Fig. 4, notice the logarithmic scale). It can be connected with the fact that the Li dipoles are merged into large clusters or even domains at these concentrations. A strong Li – Li interaction prevents their reorientation. It implies that there is energy, $w$, which is necessary to reorient dipoles. The electric field applied decreases this energy linearly. Such dependence can be seen even for low Li concentrations if the sample is strained. There can be also some differences due to different protocol in the FC procedure (for instance, due to different annealing temperatures).

Finally, we have observed a pseudolinear field dependence of dielectric stiffness in [100] PMN, PMN-PT35 and PMN-PT30 single crystals in the FC mode. We have explained this dependence by polarization fluctuations (rotations) and domain wall movements. Within the same approach we managed to explain the experimental data on the pseudolinear dependence of the SHG intensities on $E$ in KLT at small Li concentrations.

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