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ORDER, DISORDER, AND PHASE TRANSITION IN CONDENSED SYSTEM

Enhancement of the Magnetocapacitance Effect in an External Electric Field in $La_xBi_{1-x}FeO_3$ Films

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Abstract—The goal of the study is to determine the value of magnetocapacitance under the substitution of lanthanum for bismuth in thin $La_xBi_{1-x}FeO_3$ films and to find out the effect of an external bias electric field on the magnetocapacitance effect. To solve this task, the dielectric permittivity, the magnetic permeability, and the loss tangent are measured in $La_xBi_{1-x}FeO_3$ films in magnetic fields of up to 8 kOe in the range of temperatures 100 K $< T <$ 1000 K. Maxima of the permittivity and permeability at low temperatures and the dependence of the permeability on the prehistory of a sample are found. An increase in the magnetocapacitance due to the substitution of lanthanum for bismuth is observed. A giant enhancement of magnetocapacitance in an external electric bias field is revealed. These phenomena are attributed to the rearrangement of the domain structure.

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1. INTRODUCTION

$BiFeO_3$-based multiferroics [1] have been widely and intensively investigated as model objects for the study of the interaction mechanism between electric and magnetic subsystems, as well as with a view to their possible application in spin electronics [2]. The coexistence of magnetic and ferroelectric subsystems suggests the interaction between them. In magnetically and electrically ordered media, one may expect, in addition to the linear effect, higher order (quadratic, cubic) nonlinear phenomena with respect to electric and magnetic fields, as well as the switching of electric polarization by a magnetic field [3] and, conversely, the switching of magnetization by an electric field [4].

An extensive group of multiferroics is formed by media with inhomogeneous distribution of the magnetic order parameter. The spatial modulation period of sublattices in such materials may be several orders of magnitude greater than the unit cell size. In these compounds, magnetoelectric (ME) interaction is inhomogeneous [5] and is described by invariants that are linear with respect to the electric polarization. Inhomogeneous ME interaction manifests itself as spatially modulated spin structures induced by electric polarization. A modulated structure (the spin cycloid) in $BiFeO_3$ is changed as the film thickness decreases, and local magnetization arises [6]; depending on the value of elastic stresses on the film–substrate interface [7], a transition from the spin cycloid to a canted antiferromagnet with a weak ferromagnetic moment may occur for some film thickness. As a result, ferromagnetic domains can be formed due to magnetostatic interaction.

Electric polarization may result from the formation of domain walls, which can be controlled by an electric field [8]. It turns out that ferroelectric domain walls and magnetic domain walls in multiferroics are interrelated [9]. One of possible mechanisms of such a relation in multiferroics is the flexomagnetoelectric effect. A jump in electric polarization at the walls of ferroelectric domains should lead to a jump in the spatial derivative of the magnetic order parameter [10], which manifests itself in the irregularities in the magnetic structure at the domain walls. If a magnetic (antiferromagnetic) and ferroelectric domain structures coexist in a material, then the above effect can manifest itself as a pinning of magnetic domain walls at ferroelectric domain walls [11].

Small mechanical stresses may lead to the destruction of the spin cycloid [12] as a result of lifting the degeneracy in the direction of spatial modulation in the base plane. The substitution of ions with different radii for bismuth ions in $BiFeO_3$ crystal changes the magnetoelectric coupling constant [13] and gives rise to magnetization. A control voltage of 10 V rearranges the magnetic domain structure from labyrinth to stripe pattern [14]. The possibility of electric control of the

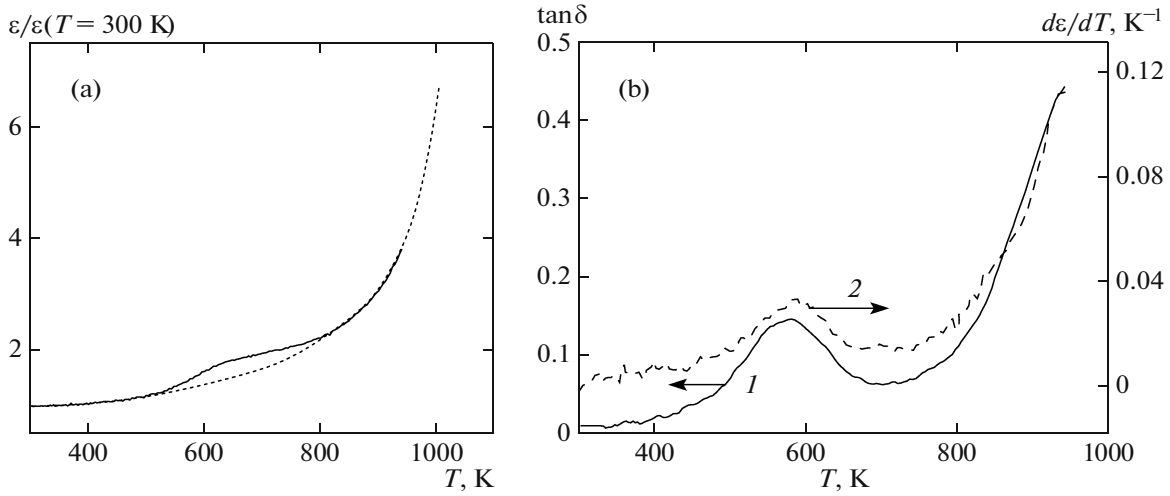

Fig. 1. (a) Dielectric permittivity normalized by the value of permittivity at $T = 300$ K and (b) loss tangent (*1*) and the temperature derivative of permittivity (*2*) for $La_xBi_{1-x}FeO_3$ with $x = 0.1$ at frequency of 10^5 Hz as a function of temperature.

magnetization of a material at room temperature is of interest from the viewpoint of its application in computer memory elements with electric recording and magnetic reading.

The goal of the present study is to determine the variation of the magnetocapacitance effect under an external bias electric field and under the substitution of lanthanum for a bismuth ion in $La_xBi_{1-x}FeO_3$ thin films and to establish a correlation between the temperature anomalies in the dielectric properties in a magnetic field and the rearrangement of the domain structure, which is observed in $BiFeO_3$ films.

2. EXPERIMENTAL RESULTS AND DISCUSSION

$La_xBi_{1-x}FeO_3$ ($x = 0.1$) films of solid solutions of bismuth ferrite were obtained by sputtering presynthesized solid solutions onto a slide by the flash method. Precursors represent powders with grain size of from 0.1 to 0.3 mm. The sputtering was performed on a UVN-71R-2 vacuum equipment for film deposition. The pressure in the reaction camera during sputtering was maintained from 10^{-3} to 10^{-2} Pa. The temperature of tantalum vaporizer was kept at about 2000°C. The substrates were placed at a distance of 10 cm from the vaporizer. The substrate temperature was varied from 250 to 300°C. The film thickness was 160 nm.

To find out the effect of the substitution of lanthanum for bismuth ions on the magnetic and dielectric properties, we investigate the permittivity and permeability in the temperature range $100 \text{ K} < T < 1000$ K. Figure 1 demonstrates the temperature dependence of permittivity normalized by the value of permittivity at $T = 300$ K and the dielectric loss tangent in $La_xBi_{1-x}FeO_3$ with $x = 0.1$. The derivative $d\varepsilon/dT$ (Fig. 1b) shows a maximum at $T_1 = 583$ K and a break at $T_1 = 835$ K. In bulk samples of $BiFeO_3$ below the Néel temperature $T_N = 646$ K ($x = 0$), the thermal expansion coefficient has a peak at $T = 533$ K and an inflection point on the temperature dependence of heat capacity at $T = 540$ K [15], which is attributed to a structural transition. The contribution due to magnetic ordering to the permittivity is determined from the approximation of the temperature dependence $\varepsilon(T)$ on the intervals 300–450 K and 850–950 K by a power-law function shown in Fig. 1a by a dotted curve. The difference of permittivities ($\varepsilon^{ex} - \varepsilon^{th}$) is attributed to the magnetoelectric contribution, which attains its maximum at $T = 640$ K in the neighborhood of the transition temperature to a magnetically ordered state. Near the ferroelectric transition, the permittivity and dielectric losses sharply increase. At $T_1 = 835$ K, there may occur changes in the crystalline structure that are associated with the transition to the orthorhombic phase.

At low temperatures, the permittivity attains its maximum at $T = 145$ K and a minimum at $T = 220$ K (Fig. 2). Dielectric losses in a $La_xBi_{1-x}FeO_3$ sample are also maximal at $T = 145$ K, and there exists an inflection point on the temperature dependence of $\tan\delta(T)$ at $T = 220$ K. Law-temperature anomaly in dielectric and structural characteristics is observed in bismuth ferrite $BiFeO_3$ nanotubes [16] and is missing in bulk samples. For instance, the slope of the temperature dependence of the frequency of Raman phonon modes is changed at $T = 140$ K. The frequency of the *E*-type phonon mode increases by 14 cm^{-1}. The dissipation of ultrasound increases, a jump in impedance (due to the increase in the capacitance) is observed, and a unit cell volume increases at $T = 150$ K. All these variations of the characteristics are

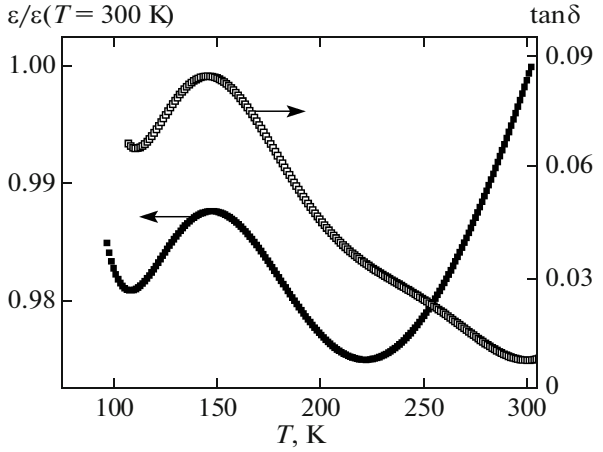

Fig. 2. Dielectric permittivity and loss tangent for $La_xBi_{1-x}FeO_3$ with $x = 0.1$ at frequency of 10^5 Hz as a function of temperature.

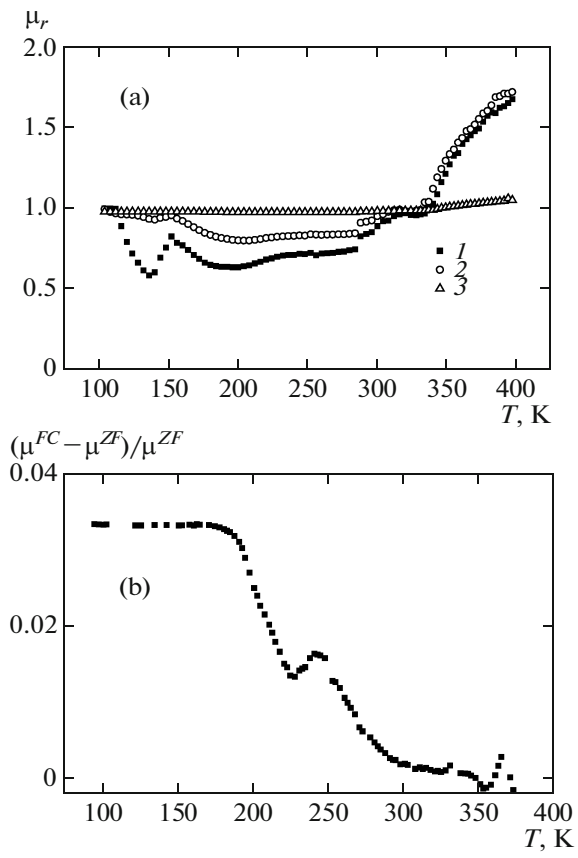

Fig. 3. (a) Magnetic permeability $\mu_r = (L_f - L_s)/L_s + 1$ of a $La_xBi_{1-x}FeO_3$ film of volume V_f in a magnetic field of $H = 2.5$ kOe at frequencies of 0.1 kHz (*1*), 1 kHz (*2*), and 10 kHz (*3*) as a function of temperature. (b) Relative variation of the permeability of the film cooled in a magnetic field of $H = 2.5$ kOe and in zero field at frequency of 10 kHz as a function of temperature.

associated with surface structural and magnetic phase transitions [17, 18]. The pyroelectric current has a sharp maximum at $T = 150$ K; this temperature is shifted to low temperatures as the sample is cooled in a magnetic field. The current is induced by electrons located in traps. The variation in the electron concentration is detected by the asymmetry of the shape of the line of an EPR signal [16]. Thus, the mechanism of the dielectric anomaly in the $La_xBi_{1-x}FeO_3$ sample is associated with the delocalization of conduction electrons in domain walls of ferroelectric and magnetic types. In the temperature range 150–200 K, electrons are localized in the vicinity of domain walls.

The magnetic permeability of the $La_xBi_{1-x}FeO_3$ film was determined from the inductance of a solenoid with inner diameter of $d = 2$ mm and length of 10 mm into which the film was placed. We measured the inductance of the coil with (L_f) and without (L_s) the sample. Since the inductance of the solenoid $L = n^2\mu\mu_0 V$ is proportional to the sample volume, the permeability μ_r of the film of volume V_f is determined as $\mu_r = (L_f - L_s)/L_s + 1$. The permeability μ_r at frequencies of 10 kHz and below (Fig. 3) has a maximum in the neighborhood of temperature of $T = 150$ K, below which a sharp increase in the Q-factor is observed (Fig. 4). The permeability in a magnetic field of $H = 2.5$ kOe exhibits a small jump at $T = 280$ K. In the vicinity of this temperature, at $T = 276$ K, the pyroelectric current in $BiFeO_3$ sharply increases in an external electric field with $U = 450$ V and is not changed in the absence of the field [16]. Further heating sharply increases the permeability.

The anomalies of the dynamic characteristics are explained by a change in the configuration of the domain structure. Ferroelectric domain walls are charged, and the charge is screened by electrons from the donor levels of the lanthanum ion. At low temperatures, electrons are localized on impurity levels and firmly hold the domain wall. The variation in the direction of domains in an external field at $T = 280$ K reduces the activation barrier associated with the interaction of domain walls with electrons and induces an electric current.

According to the calculations of the electron density functional for charged vacancies of bismuth ions, the energy of localized electron states is close to the Fermi energy [16]. This mechanism is confirmed by the measurement of the magnetic permeability of a film cooled in zero magnetic field and in a field of 2.5 kOe, as illustrated in Fig. 3b. In a magnetic field, the magnetization of a domain directed along the field increases, and the permeabilities of films under cooling in a magnetic field and in zero field are different at $T < 280$ K. A further decrease in temperature induces the localization of electrons at $T = 225$ K, which correlates with the temperature $T = 220$ K of the maximum pyroelectric current in $BiFeO_3$ films [16]. In this range of temperatures, the Q-factor of magnetic oscil-

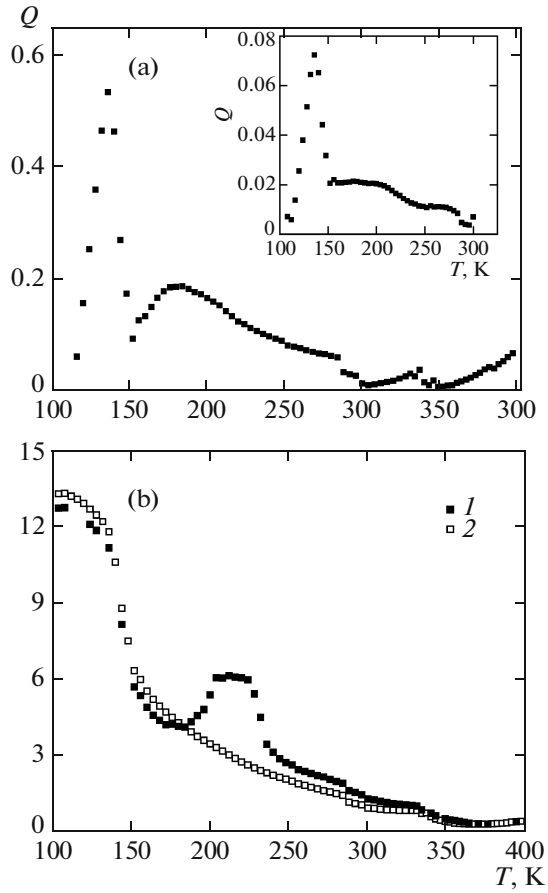

Fig. 4. (a) Q-factor of a $La_xBi_{1-x}FeO_3$ film with $x = 0.1$ at frequencies of 1 kHz (*1*) and (b) the film cooled in a magnetic field of $H = 2.5$ kOe (*1*) and in zero field (*2*) at frequency of 10 kHz as a function of temperature. Inset: the Q factor at frequency of 0.1 kHz as a function of temperature.

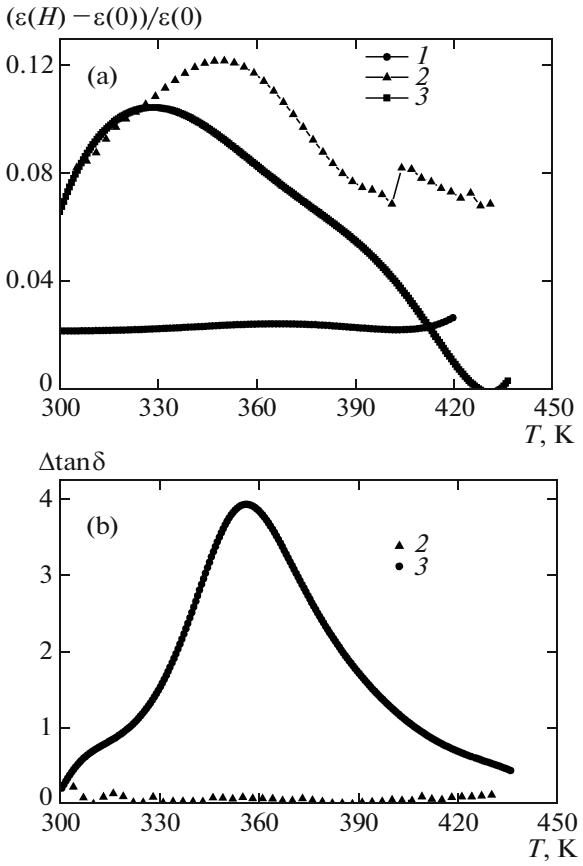

Fig. 5. (a) Magnetocapacitance of a $La_xBi_{1-x}FeO_3$ film with $x = 0.1$ at frequency of 10^5 Hz in a magnetic field of $H = 8$ kOe with a bias voltage of $U = 0$ (*1*), 0.5 V (*2*), and 1 V (*3*) as a function of temperature. (b) Relative variation of the dielectric loss tangent $\Delta\tan = (\tan\delta(H) - \tan\delta(0))/\tan\delta(0)$ with $U = 0.5$ V (*2*) and $U = 1$ V (*3*) in a magnetic field as a function of temperature.

lations also has a maximum at frequency of $\omega =$ 10 kHz when cooling in a magnetic field.

We measure the dielectric permittivity in a magnetic field of $H = 8$kOe at two frequencies, $\omega = 10$ kHz and 100 kHz, in the temperature interval 300 K $< T <$ 450 K. The magnetic field is directed parallel to the plates of the capacitor and is perpendicular to the electric field. In the absence of a bias electric field, the permittivity monotonically increases by 2.5% under heating in a magnetic field, which is greater than the magnetocapacitance of $BiFeO_3$ [15]. The magnetocapacitance of the $La_xBi_{1-x}FeO_3$ film is determined for two values of voltages $U = 0.5$ V and $U = 1$ V. In bias fields, the permittivity slightly decreases under heating, passes through a minimum at $T = 355$ K and $U =$ 0.5 V, and increases at $T = 325$ K and $U = 1$ V. The temperature dependence of magnetocapacitance is demonstrated in Fig. 5a. The magnetocapacitance $(\varepsilon(H) - \varepsilon(0))/\varepsilon(0)$ has a maximum whose magnitude is five times greater than the value of magnetocapacitance in the absence of an external electric field (Fig. 5a). As a bias electric field increases, the magnetocapacitance decreases and vanishes at $T = 430$ K in a bias field of $U = 1$ V.

The dielectric loss tangent in a film with a bias voltage of $U = 0.5$ V is almost independent of the external magnetic field and smoothly increases under heating, whereas, in a bias field of $U = 1$ V, dielectric losses are minimal at $T = 360$ K and strongly increase in an external magnetic field. The relative variation of losses with temperature in a magnetic field has a maximum and vanishes in the region of high temperatures (Fig. 5b).

The temperature dependence of magnetocapacitance shown in Fig. 6 is qualitatively the same at frequencies of $\omega = 100$ kHz and $\omega = 10$ kHz, whereas dielectric losses in a magnetic field decrease (Fig. 6b) at frequency of 10 kHz, and, as temperature increases, the effect of the magnetic field decreases and completely vanishes under the application of a bias voltage of $U = 0.5$ V. The relaxation and enhancement of

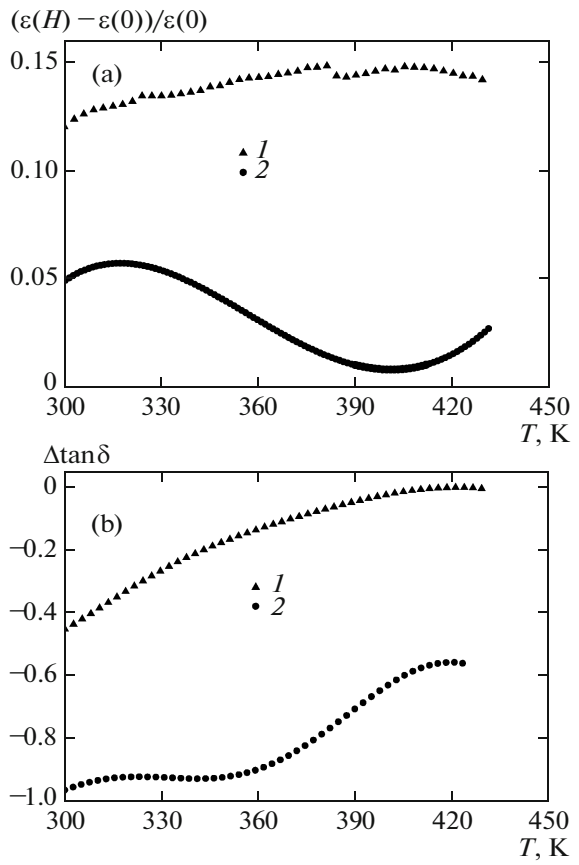

Fig. 6. (a) Magnetocapacitance of a $La_xBi_{1-x}FeO_3$ film with $x = 0.1$ at frequency of 10^4 Hz in a magnetic field of $H = 8$ kOe with a bias voltage of $U = 0.5$ V (*1*) and $U = 1$ V (*2*) as a function of temperature. (b) Relative variation of the dielectric loss tangent $\Delta\tan = (\tan\delta(H) - \tan\delta(0))/\tan\delta(0)$ with $U = 0.5$ V (*1*) and $U = 1$ V (*2*) in a magnetic field as a function of temperature.

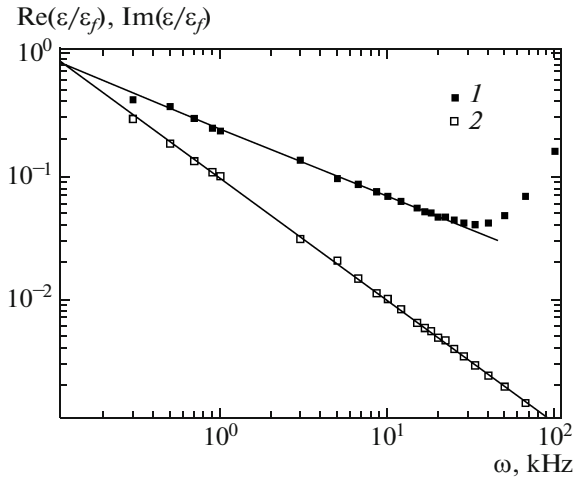

Fig. 7. Real (*1*) and imaginary (*2*) parts of the dielectric permittivity of a $La_xBi_{1-x}FeO_3$ film with $x = 0.1$ normalized by the value of permittivity at frequency of 0.1 kHz as a function of frequency at room temperature. The fitting function is $\mathrm{Im}(\varepsilon(\omega))$ / $\mathrm{Im}(\varepsilon(\omega = 100\ \mathrm{Hz})) = 100/\omega$ (solid line).

dielectric losses in a magnetic field at two frequencies is related to the relaxation mechanism, which is determined from the frequency dependence of permittivity shown in Fig. 7. The frequency dependence of the real part of permittivity is not described by the Debye model. The reason for such a discrepancy is that the Debye relation does not take into account the presence of free electric charges. The imaginary part of permittivity is described by the Debye model on the entire frequency interval 0.1 kHz < ω < 100 kHz is described by the expression: $\mathrm{Im}(\varepsilon) = \chi_d(0)(\omega\tau)/(1 + (\omega\tau)^2)$. The fitting function $\mathrm{Im}(\varepsilon(\omega))/\mathrm{Im}(\varepsilon(\omega = 100\ \mathrm{Hz})) = 100/\omega$ shown in Fig. 7 completely agrees with experimental data.

In the film investigated, there are free charges in the vicinity of domain walls. In this case, the dielectric permittivity is attributed to the effective dipole moment produced by charged domain wall and electrons. For $\omega_r < \omega$, we represent the normalized real part of permittivity as a sum of two contributions of conduction electrons and a resonance term from a domain wall in the form

$$\mathrm{Re}(\varepsilon)/\mathrm{Re}(\varepsilon(\omega = 100\ \mathrm{Hz})) = A + B/\omega^n + C/(\omega_0^2 - \omega^2). \quad (1)$$

This function with parameters $n = 0.8$ and $\omega_0 = 200$ kHz well describes the experimental data. What is the physical reason for the interaction between electric and magnetic dipole moments in $La_xBi_{1-x}FeO_3$ thin films?

The formation of a magnetic irregularity under substitution of lanthanum for bismuth gives rise to electric polarization whose spatial distribution symmetry is determined by the symmetry of the magnetic irregularity. Inhomogeneous magnetoelectric effect takes place in magnetic crystals of any symmetry. Spatial modulation of the magnetic order parameter in the material gives rise to electric polarization; in turn, the electric field changes the inhomogeneous magnetic ordering.

In bismuth ferrite $BiFeO_3$, ferroelectric polarization is directed along a cube diagonal and has eight possible orientations corresponding to positive and negative directions of the four diagonals of the cube. Under an external electric field, the ferroelectric polarization vector discretely rotates through angles of 180°, 109°, and 71° [19]. The state of polarization after the orientation transformation is stable and persists after removing the field. In thin $BiFeO_3$ films, spins are perpendicular to the rhombohedral axis [20]. Moreover, there is sixfold degeneracy in the [111] plane, which can be lifted by an external electric field; this may lead to orientational phase transitions.

The interaction between ferroelectric and magnetic domains changes the magnetic structure in an antiferromagnetic domain wall: spins leave the plane of rotation near the ferroelectric domain walls, and the anti-

ferromagnetic domain wall falls into a potential well formed by ferroelectric domains. This reduces the mobility of domain walls and changes their dynamic properties.

The existence of magnetization in the neighborhood of ferroelectric domain walls was pointed out by theoretical calculations [21, 22] and experimental investigations of $BiFeO_3$ films [23]. Magnetization of domain origin associated with the inhomogeneous magnetoelectric effect has a value of $M \sim 3$ G [24], which is comparable with the contribution to magnetization due to the Dzyaloshinskii–Moriya interaction, which is about 2 G [25]. Taking into account these factors increases the resulting magnetization in a bismuth ferrite film and leads to high sensitivity of the magnetic and ferroelectric susceptibilities to external effects. For example, the application of a coercive voltage of 5 V rotates the polarization vector through 109° or 180° with respect to the initial position [26].

Thus, the phenomena observed can be associated with domains and domain walls. Three types of domains have different coercive fields and different oscillation frequencies. We observed the rotation of ferroelectric domains in electric fields in 600-nm-thick $BiFeO_3$ films through 109° at $T > 300$ K by the method of resonant piezoelectric spectroscopy with the rotation of antiferromagnetic domains through 90°, which was determined by the photoemission electron microscopy technique with linear dichroism of X-ray radiation; the rotation of domains through 71° occurs at higher temperature of $T = 380$ K [19]. The switching time is 10^{-5} s [27].

In external fields applied at an angle to the axis of polarization or magnetization of a domain, the frequencies split. In the general case, one has a noncollinear arrangement of domains with oscillation frequency $\omega_0 = \gamma H_A - H$, where H_A is the anisotropy field and H is the external magnetic field. Magnetization and polarization also increase in external magnetic and electric fields due to the shift of domain walls and the rotation of domains, which increases the dynamic dielectric and magnetic susceptibilities:

$$\begin{aligned} \mathrm{Re}\,\chi &= \chi_0/(1+(\omega\tau)^2) + \chi_0/(\omega_0^2 - \omega^2 + \beta^2), \\ \mathrm{Im}\,\chi &= \chi_0\omega\tau/(1+(\omega\tau)^2) + \beta\chi_0/(\omega_0^2 - \omega^2 + \beta^2). \end{aligned} \quad (2)$$

The expression for the relaxation frequency of ferroelectric domains is similar to the expression for the relaxation time in a superparamagnet: $\tau = \tau_0 \exp(E_a/k_B(T - T_0))$, where T_0 is the blocking temperature of domains and E_a is the activation energy. The magnetocapacitance and the dielectric loss tangent at frequency of 100 kHz are associated with the closeness to the resonance frequency of domain walls. A typical resonance frequency is $\omega_0 \approx 10^6$ Hz, and if the resonant absorption linewidth $\beta = \Delta\omega < (\omega_0 - \omega)$ and $\mathrm{Im}\chi > 1$, then the variation of the dielectric loss tangent in a magnetic field can be expressed as $\tan\delta_H/\tan\delta_0 = \beta_H\chi_H/\beta_0\chi_0$. In a magnetic field, the activation energy decreases, $E_a(H) = E_a - \Delta E$, and the dielectric losses increase, $\beta \approx 1/\tau$, $\beta_H/\beta_0 = \exp(\Delta E/(k_B(T - T_0))) > 1$, and reach a maximum at $T_0 = 355$ K in the neighborhood of the point of domain rotation through 71°.

In the low-frequency domain, relaxation of dipole moments described by the Debye model is dominant. The Q-factor of magnetic oscillations at frequencies $\omega < 10$ kHz at $T > 288$ K becomes less than one, and the spectrum acquires a relaxation character. The relaxation frequency of a domain wall is $\omega_r = 2M_0^2/\chi da$ [28], where M_0 is the magnetization of the film, χ is static susceptibility, d is the thickness of the domain, and α is the damping coefficient, which is inversely proportional to the domain thickness. A shift of a domain wall results from the rotation of magnetization or polarization vectors. In an external electric field, the domain thickness and the susceptibility are increased; accordingly, the relaxation frequency is decreased. The variation of dielectric losses in a magnetic field in the Debye model is proportional to $\mathrm{Im}(\varepsilon(H) - \varepsilon(0))/\mathrm{Im}(\varepsilon(0)) = (\tau(0) - \tau(H))/\tau(H) = \chi(0)d(0)/\chi(H)d(H) - 1 < 0$.

Thus, the dielectric relaxation is attributed to the motion of domain walls and the switching of orientations of ferroelectric domains in the temperature interval 280–360 K in an external electric field and is described by the Debye model in the low-frequency domain. At high frequencies, near the resonance frequency of domain walls, the dielectric losses are associated with the transfer of oscillation energy of domain walls to conduction electrons in $La_xBi_{1-x}FeO_3$ films.

3. CONCLUSIONS

In the low-temperature region, we have observed maxima of magnetic permeability and electric permittivity, that are associated with the pinning of electrons at domain walls. In the temperature interval 275–285 K, the temperature derivatives of the permeability and permittivity attain maximum values associated with the rotation of domains through 109°. The dielectric relaxation is described by the Debye model at frequencies below 10 kHz. The enhancement of magnetocapacitance in an external electric field with a maximum in the temperature interval 350–380 K is attributed to the rotation of domains, observed in $BiFeO_3$ films, through 71° [19]. The decrease in dielectric losses at frequencies of about 10 kHz is due to the increase in the relaxation time of domain walls in a magnetic field in the Debye model. At higher frequencies, 100 kHz, dielectric losses increase in a magnetic field due to the decrease in the relaxation time and the activation energy of domain rotation in the range of domain switching temperatures.

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