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Dielectric and switching properties of ferroelectric superlattices and multilayers

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The study has investigated the dielectric and switching properties of the ferroelectric superlattices and multilayer formations with a barium-titanate-type structure of separate layers. The properties were studied in detail on the $BaTiO_3/BaZrO_3$ ferroelectric superlattices (16 pairs of the layers), which demonstrate a ferroelectric phase transition at the temperature of 393 °C, which significantly exceeds the temperature of the phase transition in the bulk and the thin-film $BaTiO_3$. Other objects of research were multilayer formations strontium titanate/lead titanate/strontium titanate, where materials of the layers have similar temperatures of the transition into the ferroelectric state and similar parameters of a lattice cell. We have identified differences in a behavior of the properties of the $BaTiO_3/BaZrO_3$ superlattices and the $SrTiO_3/PbTiO_3/SrTiO_3$ multilayer formations, which are related to different intensity of mechanical and electric interactions of the layers that make up these structures.

Keywords: ferroelectric superlattice, ferroelectric multilayer formation, dielectric permittivity, switching current, mechanical stress, phase transition.

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1. Introduction

Presently, the ferroelectric materials are widely used and applied in microelectronics, micromechanics, nonvolatile memory and in other applications [1–8]. In case of using homogeneous crystal or ceramic structures in practice, characteristics of the used materials are usually changed in a desirable direction by varying an elemental composition, the crystal structure (a single crystal or a polycrystal) a geometry of the device, for example, a film thickness and a material of electrodes. The multilayer materials [9,10] have a wide list of parameters that govern their characteristics. The characteristics of these structures, which are used in practice, can be significantly changed as a result of strong mutual influence of separate layers of the multilayer system, which is achieved by varying, for example, the thickness of the homogeneous layers that make up the multilayer structure, using various combinations of the composition of two or several crystalline phases, varying ratios between portions of each of them, etc.

A nature of the mutual influence of the layers of the multilayer material, which enable varying its properties in a practically desirable direction is based on variability of the properties of the said material by varying long-range elastic or electric fields that act in these structures as well as short-range chemical interactions of the components that contact in these structures [11–13]. Among the designated factors, the influence of elastic deformations for obtaining the required characteristics of the multilayer materials is now used most often.

Practical application of the ferroelectric materials in the various devices of microelectronics and nanoelectronics, in particular, in instruments and devices of nonvolatile memory, requires investigating the dielectric properties and processes of switching of polarization in the ferroelectric thin-film structures. The first stage of the present study was investigation of the influence of the factors that control changes of the parameters of the superlattice barium titanate/barium zirconate $BaTiO_3/BaZrO_3$ (BT/BZ) on its dielectric and switching properties. The studied samples consisted of 16 pairs of the parallel layers of the said materials. A grating period, i.e. a sum of the thicknesses of the two successive layers $BaZrO_3$ (BZ) and $BaTiO_3$ (BT) was 13.32 nm for this lattice. The layers BZ and BT had the thicknesses 6.65 and 6.67 nm, respectively.

Recent structural studies of the BT/BZ superlattices done using methods of X-ray diffractometry and Raman-scattering spectroscopy have shown that almost for all the periods of the ferroelectric superlattices tensioning the $BaTiO_3$ layers as a result of epitaxial conjugation with the

$BaZrO_3$ layers that have a larger cell size stabilizes layer-plane polarization in the $BaTiO_3$ layer. The bulk BT has a tetragonal structure with the sizes $a_{BT} = 3.992$ Å and $c_{BT} = 4.036$ Å while BZ has a cubic structure with the size $a_{BZ} = 4.192$ Å.

The barium zirconate itself is not a ferroelectric, but a paraelectric, i.e. it has not spontaneous polarization in a free state. It is obvious that in the superlattice in question mechanical stresses occur when two layers with different values of the lattice constant parameters are combined. In doing so, in the formed superlattice a lattice cell of barium zirconate is compressed and a lattice cell of barium titanate is stretched.

2. Dielectric and switching properties of ferroelectric superlattices barium titanate/barium zirconate

The temperature dependences of dielectric permittivity ε and a dielectric loss angle tangent $\mathrm{tg}\,\delta$ for the sample of the superlattice barium titanate/barium zirconate are shown in Figures 1 and 2.

The temperature dependence of dielectric permittivity is studied to show that the studied ferroelectric superlattice exhibits the ferroelectric phase transition at the temperature of 393 °C. We note that the found value of the temperature of the phase transition for the said superlattices is much higher as compared to pure barium titanate both in a bulk form and as thin films as well [14–16].

The value of dielectric permittivity of the $BaTiO_3/BaZrO_3$ lattice in a maximum that corresponds to the phase transition is high and exceeds the value of dielectric permittivity of barium titanate both in the bulk form and as the thin films as well. The said value is almost 11000 units in the Curie point.

The dependence of reciprocal dielectric permittivity on the temperature was constructed to show that relative to the dependence $1/\varepsilon(T)$ (Figure 3) the studied superlattice behaves as a material with the second-order phase transition.

Not only a Curie-Weiss law is met for it, but the law of „two“ is met as well: a ratio of slope angle tangents of the dependence $1/\varepsilon$ on the temperature above and below the Curie point is ~ 2.0.

With an increase of a frequency of a measurement field, the values of dielectric permittivity of the studied superlattice decrease within the entire studied temperature interval (Figure 4). At the same time, the position of the maximum on the dependence $\varepsilon(T)$ is almost unchanged with variation of the frequency.

The loops of dielectric hysteresis were studied at the various temperatures to show a change of spontaneous polarization P (Figure 5) and the coercive field E_c (Figure 6) near the Curie point as well as presence of the internal bias field E_{bs} in the created structures (Figure 7).

It is clear from Figure 5 that spontaneous polarization in the said superlattices is reduced quite slowly when

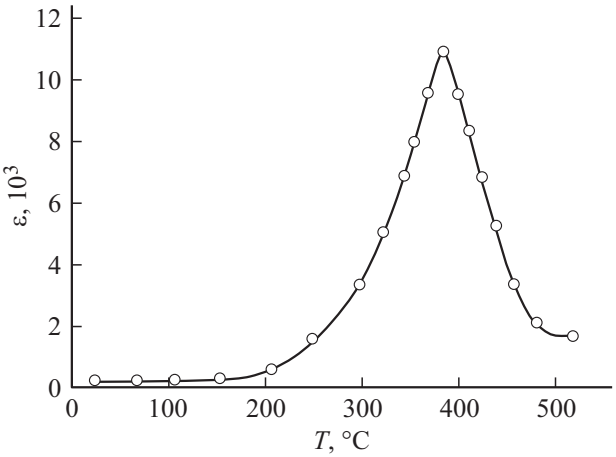


Figure 1. Dependence of dielectric permittivity on the temperature for the sample of the $BaTiO_3/BaZrO_3$ superlattice. The frequency of the measurement field is 1 kHz, while the measurement voltage is 0.1 V.

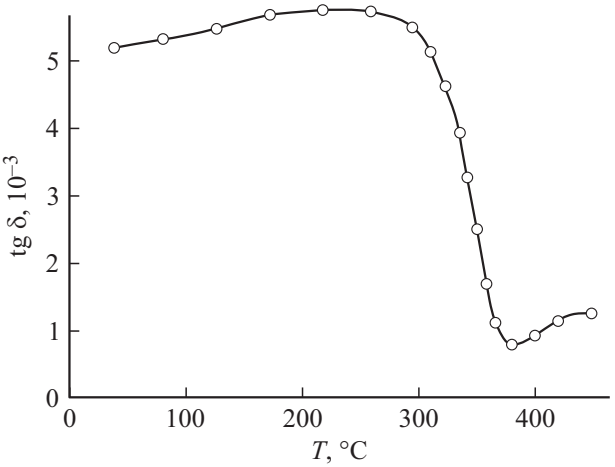


Figure 2. Temperature dependence of the dielectric loss angle tangent for the sample of the $BaTiO_3/BaZrO_3$ superlattice. The frequency of the measurement field is 1 kHz, while the measurement voltage is 0.1 V.

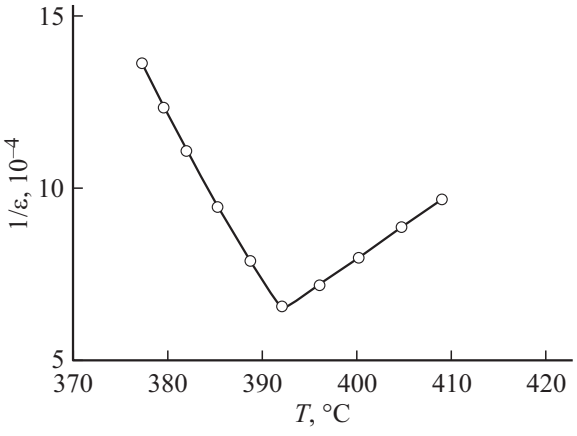


Figure 3. Dependence of dielectric permittivity on the temperature for the sample of the $BaTiO_3/BaZrO_3$ superlattice.

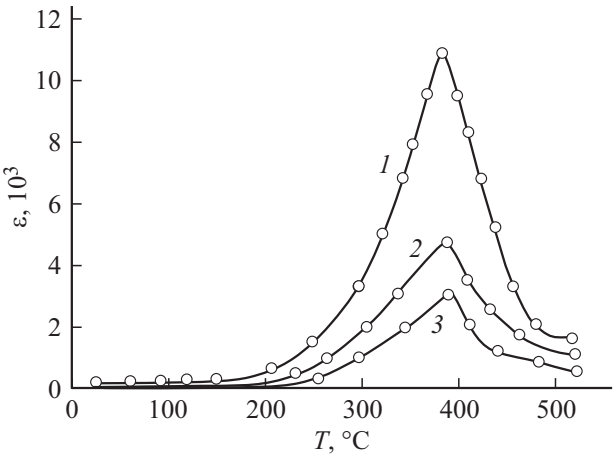


Figure 4. Temperature dependences of dielectric permittivity for the sample of the $BaTiO_3/BaZrO_3$ superlattice at the various frequencies of the measurement field, kHz: *1* — 1, *2* — 10 and *3* — 50.

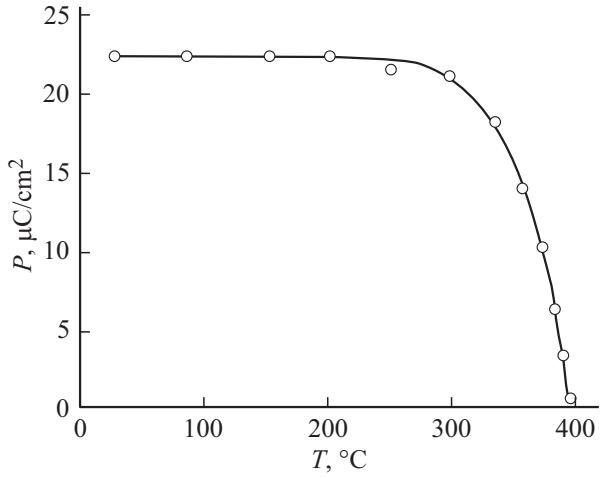


Figure 5. Temperature dependence of spontaneous polarization for the sample of the $BaTiO_3/BaZrO_3$ ferroelectric superlattice.

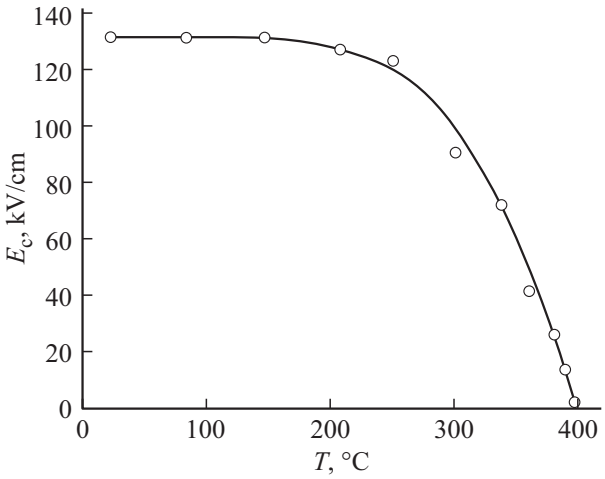


Figure 6. Temperature dependence of the coercive field for the sample of the $BaTiO_3/BaZrO_3$ ferroelectric superlattice.

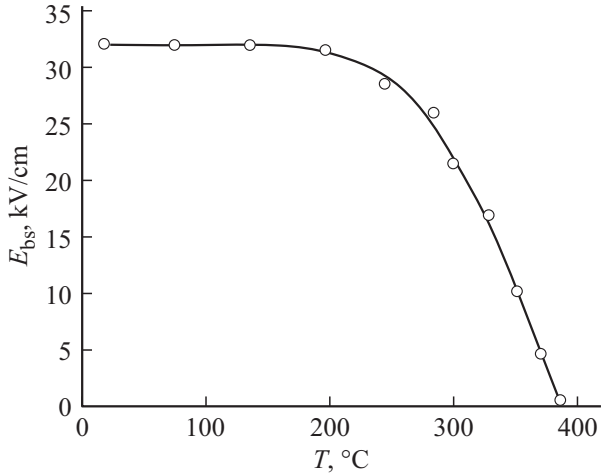


Figure 7. Temperature dependence of the internal bias field for the sample of the $BaTiO_3/BaZrO_3$ ferroelectric superlattice.

approaching the temperature of the phase transition, occupying the certain temperature interval below 200 °C.This distinguishes the superlattice in question from bulk barium titanate, in which variation of polarization when approaching the temperature of the phase transition is characterized by a sharp decrease. Spontaneous polarization of the studied superlattice turned out to be $22.5\,\mu C/cm^2$.

The coercive field of the superlattice in question was calculated to show that $E_c = 135\,kV/cm$ at the room temperature and decreases to zero, starting from the temperature of 200 °C to the temperature of the phase transition. The behavior of the dependences $P(T)$ and $E_c(T)$ as well as absence of hysteresis in the Curie point shows transformation of the phase transition from the first order for barium titanate into the second order for the studied lattice.

A direction of the internal field E_{bs} was determined in the present study by applying a constant biasing field to the studied superlattice to shift the loop of dielectric hysteresis according to a direction of the applied electric field. The loops of dielectric hysteresis (Figure 8) of the studied material were analyzed to show their asymmetry relative to the horizontal axis, i. e. to strength of the electric field.

A small shift of the loop of hysteresis along the axis of strength of the electric field means that the superlattices in question have a so-called internal bias field E_{bs} due to asymmetry of the studied structure as a result of presence of the substrate and a difference of the materials of upper and lower electrodes relative to a polar direction.

When applying the voltage to the sample with a positive pole to the substrate, the loop of hysteresis is shifted oppositely to the loop shift under effect of only the internal bias field that acts in the sample. It means that the synthesized superlattices have the internal bias field. Results of its determination show that at the temperature of

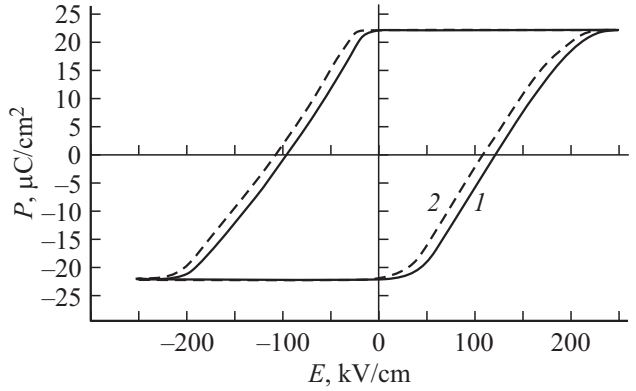


Figure 8. Loops of dielectric hysteresis for the sample of the $BaTiO_3/BaZrO_3$ superlattice at the temperature of 20 °C: *1* — without the external field (the solid curve) and *2* — with the additionally applied field (the dashed line).

20 °C the internal bias field is 32 kV/cm and insignificantly decreases with the increase of the temperature.

In order to explain causes of the experimentally-originated internal biasing field and its direction, on e can use representations about a flexoelectric effect. Let us consider a case when the lower electrode is in direct contact with a barium zirconate layer. The size of the lattice cell of the lower (near the substrate) electrode along a direction of contact is smaller than that of the barium zirconate layer that contacts the electrode. It induces compressive stresses along a direction parallel to the substrate plane, which act on a barium zirconate cell. As a result of effect of the said stresses the barium zirconate lattice cells that contact this electrode are shaped as a trapezoid, whose short base contacts the electrode. Due to this change of the form of the lattice cell, a positively-charged ferroelectrically-active zirconium ion in its center is squeezed out to a side opposite to the substrate, which exactly means that a negative charge opposite to it appears at a boundary with the electrode.

If the barium titanate layer contacts the electrode, then the considered trapezoidal change of the shape of the lattice cells is transferred through it to the next barium zirconate layer. At the same time, flexoelectric distortions of the barium zirconate cell, which are active in origination of the internal field, will be smaller, meaning that the internal field originating due to them will be smaller, too.

3. Repolarization of the ferroelectric $BaZrO_3/BaTiO_3$ superlattices

Patterns of switching the synthesized superlattices were identified in the present study by using a modified Sawyer-Tower circuit for investigating loops of dielectric hysteresis with conductivity compensation as well as a Merz method of recording switching currents that originate under effect of bipolar rectangular pulses of the electric field.

The switching current was measured by voltage drop across a resistance serially connected to the sample and recorded at a screen of a two-channel digital oscilloscope TDS 2075. For this purpose, equidistant bipolar rectangular periodic pulses of voltage that had the same duration were supplied to the samples. To do this, we used a signal generator Waveform Generator 2571 that generated the pulses with a time of an increase of the switching voltage that did not exceed 10 ns.

The switching current was found by subtracting a contribution by a discharge of a linear component of capacitance from the total current pulse. The measured values were integral switching characteristics: the maximum value of the pulse of the switching current I_{max} and the total switching time τ. The switching time τ was determined as an interval between a start of the current pulse and a time when the value of the switching current decreased to 10% of the maximum value I_{max}.

The switching current pulses of the studied superlattice for the various values of the switching fields are shown in Figure 9.

The experimentally-obtained dependence of the switching current on the applied field E (Figure 10) is well known when describing repolarization of the ferroelectrics [1,2].

Its inflection point divides an activation portion (the area of the „weak“ field, where domains originate and the current dependence on the field is described by an exponential law $1/\tau \sim \exp(-\alpha/E)$, α is the activation field) and a domain growth portion (the area of the „strong“ fields — a linear portion of the said dependence).

A boundary between the areas of the activation and nonactivation switching modes, — the so-called threshold or critical field E_{th} that is determined by the switching currents, approximately corresponds to the coercive field that is determined by the loop of dielectric hysteresis. The

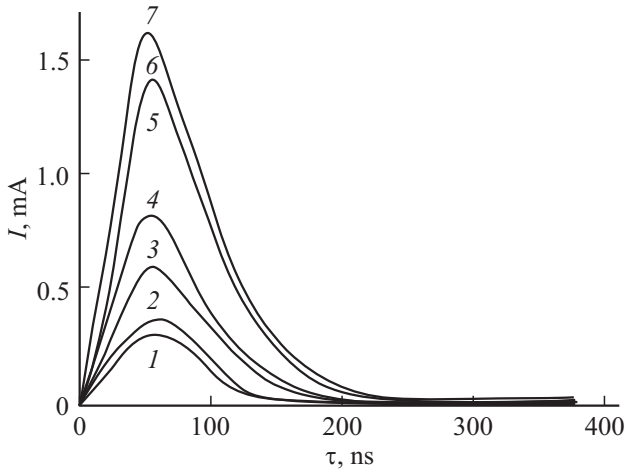


Figure 9. Pulses of the switching currents of the $BaTiO_3/BaZrO_3$ superlattices for the various values of the switching fields E, kV/cm: *1* — 22, *2* — 28, *3* — 117, *4* — 164, *5* — 187, *6* — 210 and *7* — 235.

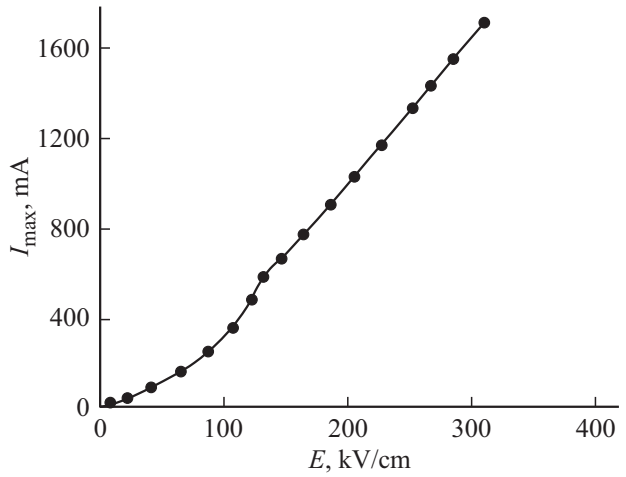


Figure 10. Dependence of the switching current on strength of the external electric field for the sample of the superlattice barium titanate/barium zirconate at the temperature of 100 °C.

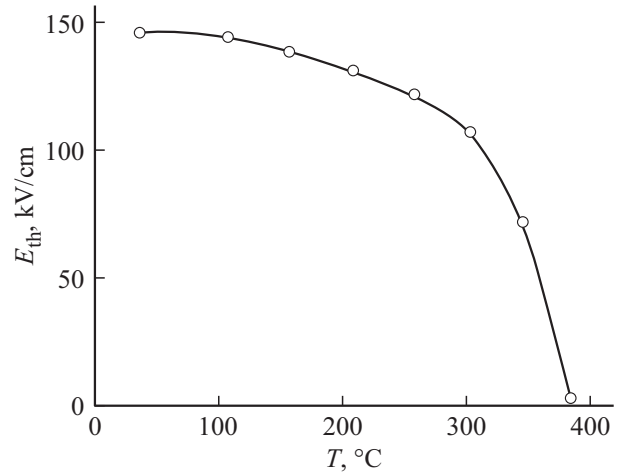


Figure 11. Temperature dependence of a threshold field that separates regions of the weak and strong fields by the switching currents for the sample of the superlattice barium titanate/barium zirconate.

threshold field decreases when approaching the temperature of the ferroelectric phase transition (Figure 11).

Figure 12 shows the experimental dependences of the switching current on strength of the external field for the sample of the superlattice barium zirconate/barium titanate within the wide temperature range.

The experimental dependences were plotted in Figure 12 in the coordinates $\ln I_{max}$ on reciprocal strength of the field $1/E$ (Figure 13) to show that for the sample of the $BaTiO_3/BaZrO_3$ superlattice the dependences of the switching current logarithm on the reciprocal field were really nonlinear. In this case they can be described within the wide temperature interval by a more complex formula $I_{max} \sim \exp(-\alpha/E^{\gamma})$ with an exponent γ for the applied field.

Values of the exponent γ for the superlattice barium titanate/barium zirconate within the weak fields at the various temperatures

γ	0.21	0.21	0.20	0.20	0.196	0.19	0.19
T, °C	24	65	120	200	250	300	350

The table shows values of the exponent γ at the various temperatures.

The table is analyzed to show that as compared to the individual ferroelectric films for the ferroelectric superlattices the said exponent has a much smaller value and is almost independent of the temperature.

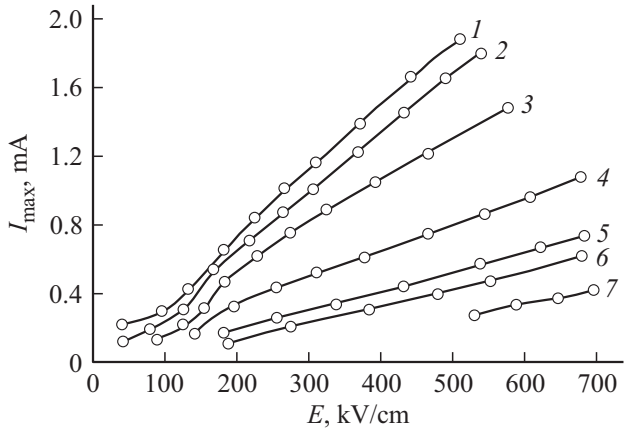


Figure 12. Dependences of the switching currents on strength of the external field for the sample of the superlattice barium titanate/barium zirconate at the various temperatures T, °C: *1* — 20, *2* — 100, *3* — 200, *4* — 250, *5* — 300, *6* — 350 and *7* — 400.

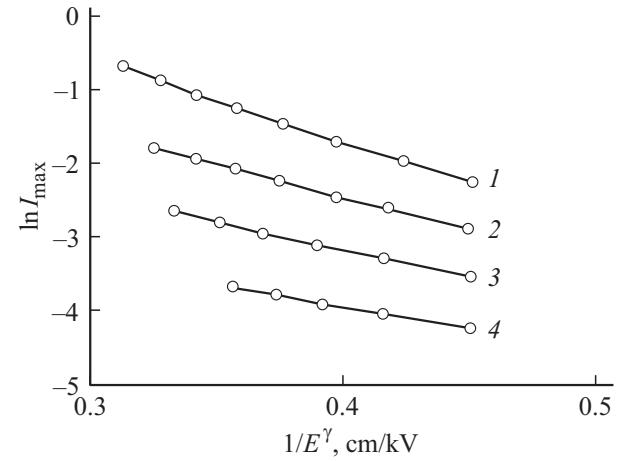


Figure 13. Dependences of a logarithm of the maximum switching current on reciprocal strength of the field, which is raised to the γ power, for the sample of the superlattice barium titanate/barium zirconate within the weak fields at the various temperatures T, °C: *1* — 65, *2* — 120, *3* — 230 and *4* — 350.

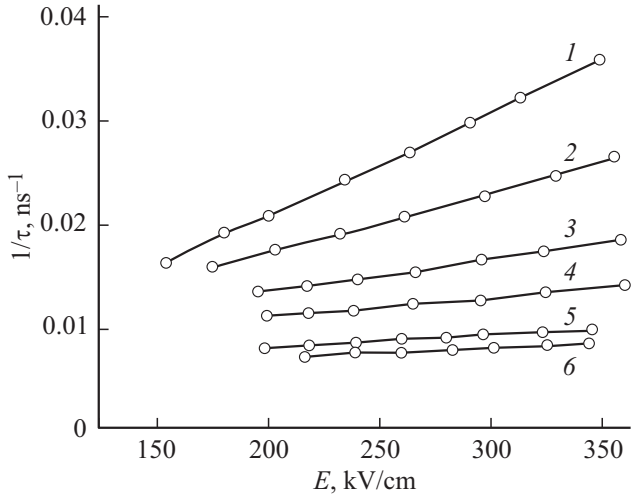


Figure 14. Dependences of the reciprocal switching time on strength of the applied field for the sample of the $BaTiO_3/BaZrO_3$ superlattice at the various temperatures T, °C: *1* — 100, *2* — 150, *3* — 200, *4* — 250, *5* — 300 and *6* — 350.

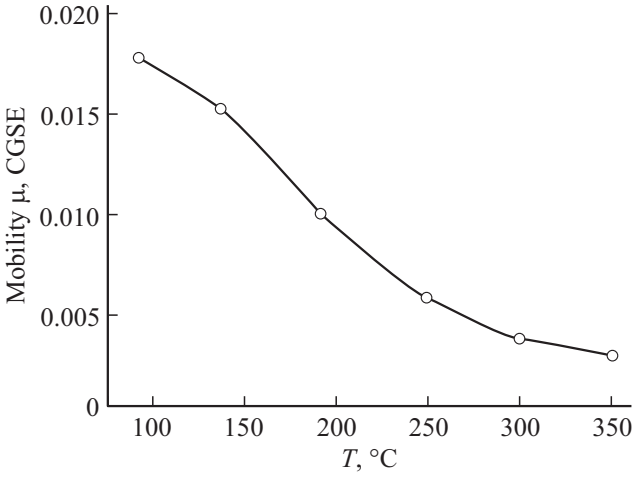


Figure 15. Dependence of mobility of domain walls on the temperature for the sample of the $BaTiO_3/BaZrO_3$ superlattice.

Figure 14 shows the dependences of the reciprocal switching time $\tau^{-1}(E)$ for the studied superlattice at the various temperatures.

Knowing the switching time, one can evaluate mobility of the domain walls μ, which is constant within the strong fields. Mobility of domain boundaries μ can be related to the switching time τ with the following relationship:

$$\mu = d/E\tau, \qquad (1)$$

where E — strength of the external electric field, d — the thickness of the sample, τ — the switching time.

Figure 15 shows the dependence of mobility of the domain walls μ on the temperature, which is obtained from processing the experimental dependences of the switching time.

It is clear that with the increase of the temperature mobility of the domain boundaries decreases. It seems to be related to the fact that with the increase of the temperature, when it approaches the temperature of the phase transition, the switching time τ increases.

By summarizing the first part of the present study dedicated to investigating the dielectric properties and the processes of switching the $BaTiO_3/BaZrO_3$ superlattices, we see that the processes of switching the superlattices in question include two stages — the activation stage and the stage of sliding of the domain boundaries. In the fields that are less than the coercive one, a dielectric response in them is mainly contributed by side motion of the domain walls, which is carried out by formation of nuclei of reverse domains on side surfaces of the domain walls and their subsequent proliferation.

Formation of the nuclei on the domain walls is related to their transitions into adjacent valleys of the potential Peierls relief (a coordinate dependence of energy (Figure 16) of the domain boundaries in a discrete lattice).

The said transitions are realized by activation of walls of its profile by thermal oscillations of the crystal lattice. In the fields that exceed E_c, the domain boundaries move in a nonactivation method, i. e. in a sliding mode, where $I_{\max} \approx \text{const}\, E$.

The integral switching characteristics in the $BaTiO_3/BaZrO_3$ superlattices do not obey a strictly exponential dependence on field strength. It results in introduction of a dynamic indicator γ for the exponential

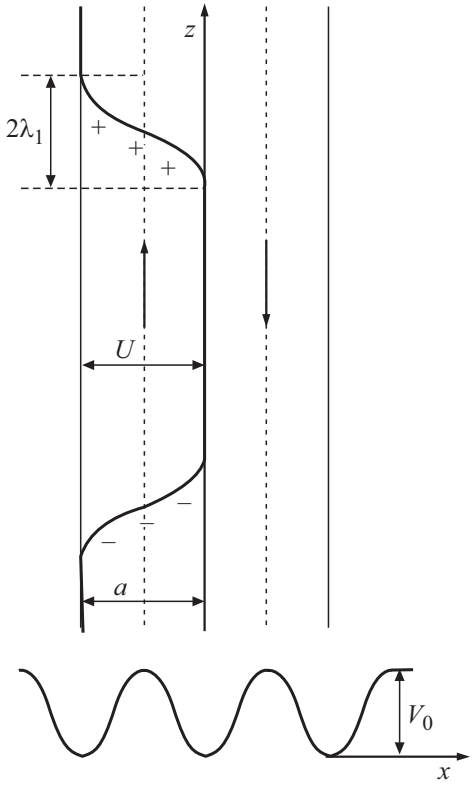


Figure 16. Origination of the charged side walls of the nucleus on the domain wall with its transition from one valley of the lattice relief into the adjacent one.

function of the switching current (a speed of motion of the domain boundaries) on strength of the electric field. The value of the said indicator in the superlattices is much smaller as compared to the thin ferroelectric films and slightly varies when approaching the point of the phase transition.

4. Dielectric properties and repolarization of the $SrTiO_3/PbTiO_3/SrTiO_3$ multilayer formations

The second task of the present study was to investigate dielectric properties of the three-layer structures strontium titanate/lead titanate/strontium titanate $SrTiO_3/PbTiO_3/SrTiO_3$ on the St-Nb substrate with a sublayer in the form of a conducting oxide $La_{1/2}Sr_{1/2}CoO_3$ as the lower electrode and platinum as the upper electrode.

The epitaxial layers $SrTiO_3$ and $PbTiO_3$ were grown on substrates separated by the $La_{1/2}Sr_{1/2}CoO_3$ oxide conducting layer of the thickness of 3.805 Å. The studied samples were grown by pulsed laser deposition using an excimer laser Lambda Physik 248 nm in the chamber MECA 2000. The $La_{1/2}Sr_{1/2}CoO_3$ buffer layer of the thickness of 50 Å was deposited at the substrate temperature of 750 °C and oxygen partial pressure of 0.2 mbarg. During deposition of the layers $SrTiO_3$ and $PbTiO_3$, the temperature and pressure of oxygen were 750 °C and 0.1 mbarg, respectively. A quality of the layer surface was examined by means of diffraction of reflected fast electrons. The thickness of the layers of strontium titanate $SrTiO_3$ and lead titanate $PbTiO_3$ was 3.5 and 4 nm, respectively. Strontium titanate $SrTiO_3$ is a so-called virtual ferroelectric with a cubic crystal lattice with a cell parameter $a = 3.905$ Å. We note that this value almost coincides with the value of the parameter $a = 3.904$ Å of the crystal cell of the $PbTiO_3$ tetragonal lattice (the lattice parameter $c = 4.150$ Å). A direction of polarization in the ferroelectric phase of this laminar structure was oriented perpendicular to the sample plane.

The temperature dependence of dielectric permittivity was measured using an impedance analyzer „Solartron 1260“ when applying the measurement field of 50 mV at the frequency of 1 kHz. The samples were placed in a resistive heating furnace, whose temperature varied from 20 to 600 °C.

Results of measurements of the temperature dependence of dielectric permittivity (Figure 17) show presence of a maximum at the temperature of 544 °C, which is considered to be a result of the ferroelectric phase transition.

When heating and cooling the studied samples, there is temperature hysteresis near 15 °C, which is typical for the first-order phase transitions. Above the temperature of the phase transition, dielectric permittivity obeys the Curie-Weiss law with the Curie constant of $4.7 \cdot 10^4$ K and the Curie-Weiss temperature of 530 °C. The ratio of the slope angle tangents of reciprocal dielectric permittivity on the

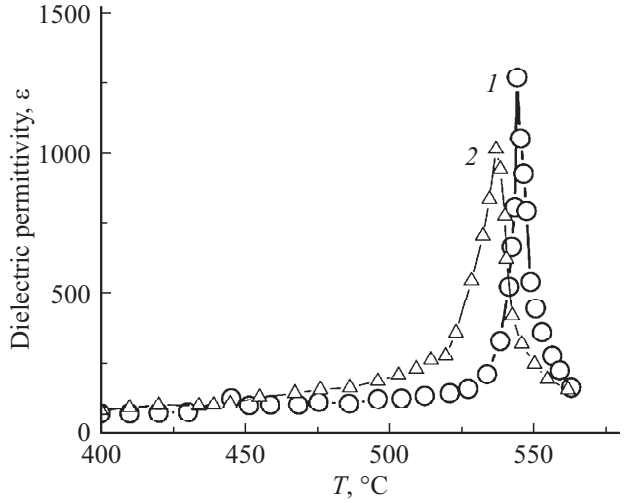


Figure 17. Dependence of dielectric permittivity on the temperature for the $SrTiO_3/PbTiO_3/SrTiO_3$ laminar structure: *1* — heating and *2* — cooling.

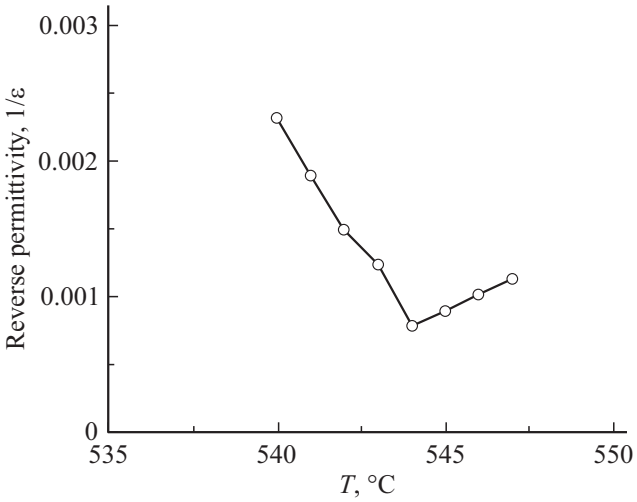


Figure 18. Dependence of reciprocal dielectric permittivity on the temperature for the sample of the $SrTiO_3/PbTiO_3/SrTiO_3$ laminar structure near the temperature of the phase transition.

temperature (Figure 18) above and below the temperature of the transition is 4.5. It also confirms that the phase transition in the studied laminar structure belongs to the first order.

As follows from the performed measurements, the $SrTiO_3/PbTiO_3/SrTiO_3$ laminar structure has the temperature of the phase transition that significantly exceeds (almost by 50 °C) the temperature of the phase transition for bulk lead titanate $PbTiO_3$. At the same time, the type of the phase transition is still the same. This change of the temperature can be induced, on the one hand, by a relatively small thickness of the very laminar structure and the ferroelectrically-active layer. With the decrease of the thickness of the sample, the Curie point is shifted toward the high temperatures. On the other hand, shifting

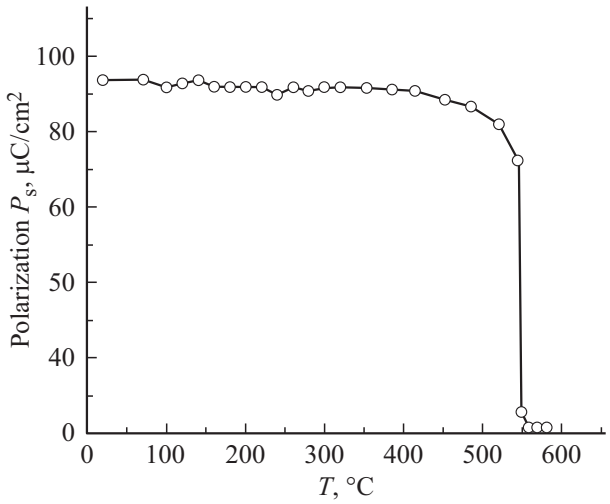


Figure 19. Temperature dependence of spontaneous polarization for the sample of the $SrTiO_3/PbTiO_3/SrTiO_3$ laminar structure.

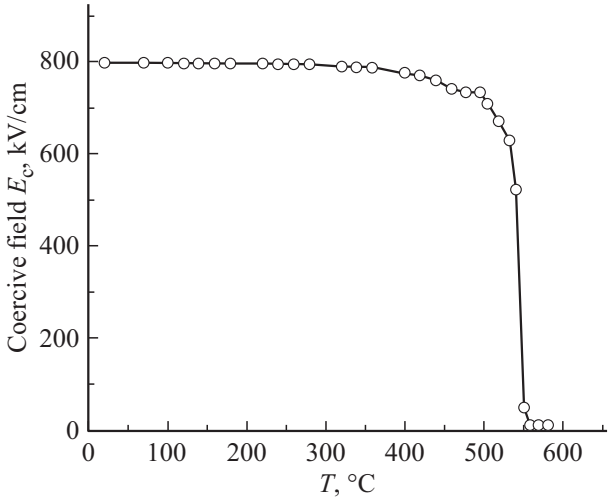


Figure 20. Dependence of the coercive field on the temperature for the sample of the $SrTiO_3/PbTiO_3/SrTiO_3$ laminar structure.

of the Curie point of the studied laminar structures into the high-temperature range can be related to origination of mechanical stress in the $PbTiO_3$ crystal lattice in a plane of joining to the $SrTiO_3$ layer. The said stress originates, first of all, due to incompliance of the parameters of the crystal cells (although it is small) and, secondly, due to incompliance of the coefficients of thermal expansion of the mating materials. When heating the sample to the high temperatures, the said factors seem to result in preservation of the polar state of the $SrTiO_3/PbTiO_3/SrTiO_3$ structure to the higher temperatures as compared to the $PbTiO_3$ ferroelectric.

In order to study the process of repolarization in the $SrTiO_3/PbTiO_3/SrTiO_3$ structures, we have studied the loops of dielectric hysteresis when applying the external sinusoidal electric field. The measurements were carried out using the Sawyer-Tower circuit for the thin-film materials with conductivity compensation. The loops of dielectric hysteresis were obtained with variation of the temperature. According to them, we have identified the temperature dependences of spontaneous polarization $P_s(T)$ (Figure 19) and the coercive field $E_c(T)$ (Figure 20) within the temperature range from 20 to 550 °C.

The dependences of polarization and the coercive field on the temperature for the studied multilayer formation are also characterized by a jump-like decrease near the temperature of the structural phase transition, which is typical for the first-order transformations.

5. Conclusion

According to the performed studies, a key factor that affects both the switching properties of the considered ferroelectric superlattices and their dielectric response is mechanical deformations that change a structure of separate layers of the superlattices and originate in them due to the difference of the sizes of the lattice cells making up the lattice of the crystal layers [17–22]. The influence of the said interactions results in the significant increase of the temperature of the phase transition into the ferroelectric state and the change of the order of the said phase transition from the first order into the second one in the $BaTiO_3/BaZrO_3$ ferroelectric superlattices.

At the same time, interaction and mutual influence of the layers $SrTiO_3$ and $PbTiO_3$ is almost minimized in the $SrTiO_3/PbTiO_3/SrTiO_3$ multilayer formations. It is, first of all, related to the fact that the sizes of the lattice cells of the mating components are almost the same, which means that mutual mechanical influence of the various layers is minimized. As a result, the $SrTiO_3/PbTiO_3/SrTiO_3$ structures preserve the first-order phase transition with hysteresis in the Curie point that is inherent to it. Besides, the jump-like behavior of the critical thermodynamic characteristics, i.e. spontaneous polarization and the coercive field, is preserved. The small layer thickness of the layer of lead titanate $PbTiO_3$ in the combined structure with the layer of strontium titanate $SrTiO_3$ results in the Curie point of the multilayer formation that significantly exceeds the Curie point of lead titanate.

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Conflict of interest

The authors declare that they have no conflict of interest.

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