

On the Electret Effect in Polymer–Ferroelectric Piezoceramic Composites with Various Values of the Electronegativity of the Polymer Matrix and Piezophase Cations

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Abstract—The influence of the electronegativity of phases, which is controlled by composite crystallization under the conditions of the effect of the electric discharge and covalence of piezophase cations, on the formation mechanism of the stable electret effect is determined. The specific features of the formation of the electret effect in composites based on polyolefins (HDPE, PP), fluorine-containing polymers (F42), and ferroelectric piezoelectric ceramics of the family of lead zirconate–titanate ($\text{Pb}(\text{Zr,Ti})\text{O}_3$) crystallized under conditions of the effect of electric discharge plasma, are revealed. A physical model of electret composites taking into account the role of homocharges and heterocharges formed in a composite by its dispersion with piezoceramic particles of various structures—rhombohedral, tetragonal, and heterogeneous—is proposed.

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INTRODUCTION

Currently, there are mainly four directions in the field of the development of electret and piezoelectric materials based on solid insulators and semiconductors. One of them is associated with the synthesis of ever new piezoceramic materials based on barium titanate and multicomponent ferroactive solid solutions of the lead zirconate–titanate family (LZT) [1–4]. The second one is based on the synthesis of temperature-resistant polar and nonpolar polymers that manifest electret properties [5–8]. A new possibility opens up a third direction, which is associated with the development of various electret composite materials based on polar and nonpolar polymers (a matrix) and microscale ferroelectric piezoelectric ceramics or semiconductor particles (disperser) [9–16]. Recently, along with the third direction, the fourth one is intensively developing, connected with the creation of matrix nanostructured electret composites [17–22]. The multiaspect character of nanotechnologies and the special properties of a nanoscale inorganic phase and polymer matrix surrounding nanoparticles make nanocomposites very important materials for different applications [19, 21, 22]. A noticeable decrease in the mobility of macromolecules in a bulk, in which the effect of micro- and nanoscale particles is spread, noticeably broadens the limit of temperature stability of the electret characteristics of the mentioned composites. We note that the unresolved and rather complex problem of the fabrication of nanoscale ferroelectric piezoelectric particles from existing efficient fer-

roelectric piezoceramics of various structures and their immobilization stands in the way of development of the technology of fabrication of electret composites with a nanoscale phase [21, 22]. Therefore, at present the most efficient direction in the development of active electret composites is still the fabrication of composite SMART materials with a microscale ferroelectric phase [15, 16, 18, 22, 23].

The aim of our study is to determine the influence of the electronegativity of phases controlled by composite crystallization under conditions of the effect of electric discharge plasma on the formation mechanism of a stable electret effect.

The aforementioned questions are solved by means of the development of technology for the fabrication of composite piezoelectrics and electrets with deep charge-capture centers by means of their crystallization under conditions of the simultaneous effect of the barrier electric discharge plasma and temperature, as well as the creation of a physical model of composites taking into account the role of homocharges and heterocharges formed in a composite by its dispersion with piezoceramic particles of various structures—rhombohedral, tetragonal, and heterogeneous.

2. EXPERIMENTAL

We considered matrix composites (0-3 type) based on piezoceramics of the lead zirconate–titanate family of the PbTiO_3 – PbZrO_3 – $\text{PbNb}_{2/3}\text{Zn}_{1/3}\text{O}_3$ – $\text{PbNb}_{2/3}\text{Mn}_{1/3}\text{O}_3$ type, polyolefins (HDPE, PP), and

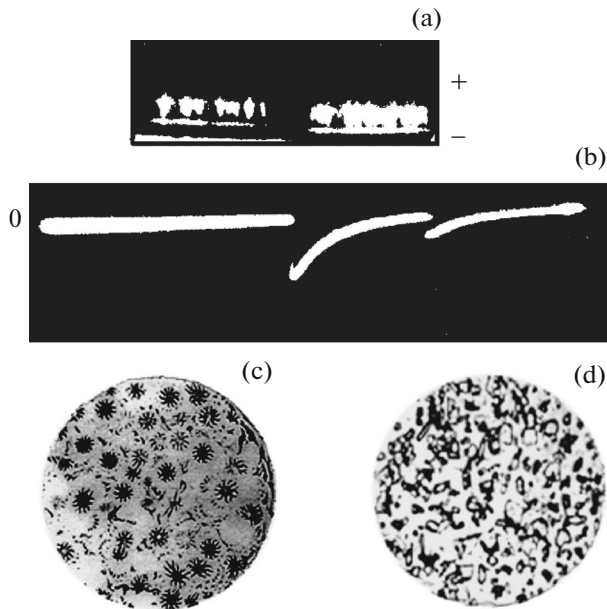


Fig. 1. Varieties of electric discharge patterns in the metal–insulator–gas–composite–metal system: (a) the optical pattern (the EOP-gram), (b) the electric (oscillogram) pattern, (c) the electrographic surface pattern of the electret composite subjected to the electric discharge effect, and (d) the microscopic surface pattern of the electret composite subjected to the electric-discharge effect.

halogen-containing polymers (F-42) [4, 6, 15]. The selection of carbochain polymers is caused by the fact that they have a high reproducibility in terms of their composition, structure, and physicochemical properties. The composite elements are fabricated by hot compaction. An electret element in the form of disc and cylinders of various geometric sizes is formed from a mechanical mixture of powders. The temperature and pressure regions of formation for composites based on halogen-containing polymers and polyolefins were (420–550) K and (30–100) MPa, respectively. The compaction time after attaining the selected temperature was 0.5 h.

The composite samples were polarized in air upon applying constant electric field with a strength (E_{pol}) from 1 to 6 MV/m at a polarization temperature (T_{pol}) of 353–450 K and time (t_{pol}) of 0.5 h. The investigation procedure of the electret effect is associated with determination of the surface charge density, sign, and its conservation duration in time [5, 8, 12]. The surface effective charge density (Q_{eff}) and charge relaxation time (τ) are determined based on the induction method with compensation of the electret potential difference U_{dif} [7]. A measuring electrode (ME), on which the charge from the charge electret surface is induced, is periodically screened using a four-blade impeller rotating in the gap between the electret and electrode. The composite electret elements were modified under the effect of electric discharge plasma

effect in air confined by insulators (partial discharge) [8, 12, 14, 16]. The modification procedures of the composite phases under conditions of the effect of electric discharge plasma are described in detail in [8, 12, 14]. To modify the composites under conditions of the effect of electric discharge plasma, we used the following devices: a UPU-10 high-voltage source ($U_{\text{out}} = 10$ kV), an N 307/1 two-coordinate signal recorder, an S1-176 two-channel oscilloscope, a U5-11 electrometer amplifier, and a V7-57.1 voltmeter. The selection of the partial discharge is due to the possibility of performing uniform surface treatment of the polymer and filler particles (Figs. 1a–1d). The electric discharge appears in the metal–insulator–gas medium–studied composite–metal system. The discharge intensity was varied by changing the amplitude of the voltage with a frequency of 50 Hz applied to the test cell. The treatment time was varied up to 2 h. The discharge power for a sinusoidal voltage period was determined by the method of the volt–coulomb characteristic [24]. The energy of separate partial discharges was determined from the optical pattern formed by an electron-optical converter (Fig. 1a) [25].

3. EXPERIMENTAL RESULTS AND DISCUSSION

Figure 1 shows the optical (Fig. 1a) and electrical (Fig. 1b) discharge development patterns in the metal–insulator–gas medium–studied composite–metal (MIGCM) system. It is seen from Fig. 1 that the electric discharge appears in the form of a series of partial discharges discrete in time and space (Fig. 1a). Each series of microdischarges corresponds to one discharge current pulse in the oscillogram (Fig. 1b). The current pulses are recorded in the presented pattern for 1/4 period of the applied sinusoidal voltage. The oscilloscope switch-on for the detection of discharge current pulses is performed upon passage of the applied sinusoidal voltage through zero (Fig. 1b). Figure 1c shows an electrographic pattern of the contact of the composite surface with discrete discharge channels. After electrographic processing, we derive the distribution pattern of microdischarges over the entire area of the composite under study. This pattern also gives information on the presence of charges formed at the places of microdischarge–composite contact. The mentioned optical, electrical, and electrographic patterns also give information on the rather uniform modification of composite elements under conditions of the effect of partial discharges in a gas gap confined with piezoelectric composites.

Figure 1d shows the microscopic surface pattern of a piezoelectric cell that has undergone discharge modification. An optical pattern of charge development is formed using an electron-optical converter developed by Academician Zavoiskii and his school [25]. The results of investigation of the electret potential difference U_{epd} of the composites depending on the struc-

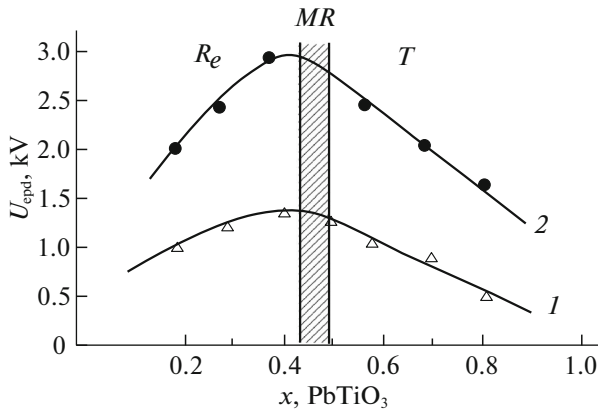


Fig. 2. Variation in U_{epd} of the PP–Pb(Ti_xZr_{1-x})O₃ composite depending on the parameter (x) of the Pb(Ti_xZr_{1-x})O₃ composition; $x = 0.2–0.8$. (1) Electrothermally polarized composites and (2) preliminarily plasma-crystallized and then electrothermally polarized composites.

ture of the piezophase are compared in Fig. 2 (curves 1 and 2). Curve 1 in Fig. 2 shows the dependence of U_{epd} on the PbTiO₃ concentration for electrothermally polarized composites, and curve 2—for preliminarily plasma-crystallized and then electrothermally polarized composites. It is seen from Fig. 2 that the regularity of varying the electret potential difference U_{epd} depending on the concentration (or the composition) of the PbTiO₃ solid solution is almost identical, but the numerical values of U_{epd} for each composition differ noticeably, i.e., plasma-crystallized electret composites possess high U_{epd} (of the electret charge). The dependence of the electret potential difference on the PbTiO₃ concentration of the piezophase has an extreme character; herewith, the maximum of this dependence is situated in the rhombohedral region near the morphotropic one. The extreme character of the dependence of U_{epd} on the PbTiO₃ concentration of the piezophase of the lead zirconate–titanate family is apparently associated with a difference of ferroelectric hardness and ferroelectric softness of the used piezoelectric ceramics. In addition, the electronegativity of cations of piezoelectric ceramic samples with the rhombohedral structure is also of importance. The presence of local mechanical stress appearing during the polarization of composites is also of no small interest. The mechanical factor is pronounced more clearly for tetragonal ceramic samples [4] and, consequently, is accompanied by a decrease in the electret potential difference. The index of increasing electronegativity of a polymer matrix under conditions of the effect of electric discharge plasma is the appearance of the following oxygen-containing groups in the IR spectrum (Fig. 3): C–O, C–O–C, OH, and CH₂. Similar results were found for composites based on fluorine-containing polymers, in

particular, PVDF [16]. Our results show that plasma modification results in strong oxidation of the polymer chain, i.e., macromolecules acquire electronegativity and a certain dipole moment. Oxidized centers serve as traps for injected charges during electrothermal polarization. The high electronegativity of oxygen-containing groups forms deep traps for charge carriers at the phase interface. In addition to the mentioned effects, macromolecules link via oxygen bridges, which is in turn accompanied by a decrease in the mobility of macromolecules and degree of freedom. The acquisition of electronegativity and the dipole moment by a macromolecule also enhances intermolecular interactions. In addition to intermolecular interaction, it is noteworthy that the oxidation of macromolecules is accompanied by an increase in interphase interactions. The use of fluorine-containing polymers as a polymer matrix is of special value because the fluorine-containing polymer is more sensitive to plasma modification. This phenomenon is caused by the high electronegativity of fluorine atoms [12]. Our problem includes the question on the influence of plasma crystallization on the microscopic parameters of the piezophase and macroscopic composite parameters in general. We used δ (the uniform parameter of spontaneous deformation) and η (the degree of domain reorientations) as microscopic parameters of the piezophase [4]. The parameters U_{epd} , ϵ , and Q_{eff} are used as macroscopic composite parameters. Figure 4 shows the dependences of ϵ , δ , and η on the PbTiO₃ concentration in TsTS-21 piezoceramics. The dependence of U_{epd} for the PP + 5% PbTiO₃–PbZrO₃ composite on the PbTiO₃ concentration in the piezophase is also presented in Fig. 4. It is seen that the maximal value of this dependence of U_{epd} is situated in the middle of a morphotropic region, where the structure of the piezophase is heterogeneous ($R_e + T$). The maximum of U_{epd} for plasma-crystallized composites is larger in terms of magnitude, and the maximum of the dependence of U_{epd} on the PbTiO₃ concentration is arranged in the tetragonal region. It is seen that the maximum of ϵ_{33}^T occurs in the morphotropic region and is displaced to the boundary of the tetragonal composition. The magnitudes ϵ/ϵ_0 and $\epsilon_{33}^T/\epsilon_0$ are compared in Fig. 4. It is seen that the amplitude of the ϵ/ϵ_0 peak (curve 4, unpolarized ceramics) is lower than in the similar dependence $\epsilon_{33}^T/\epsilon_0$ (curve 5) for the polarized ceramic samples, and is displaced towards the rhombohedral composition [4]. The results of investigation of the permittivity of the PP + 5% PbTiO₃–PbZrO₃ composite polarized at a temperature higher than the Curie temperature are of interest (curve 6). It is seen that composites polarized at temperatures higher than or equal to the Curie temperature of the piezophase have a permittivity larger in terms of amplitude, and its peak is unobservable in the region of the tetragonal structure. The vari-

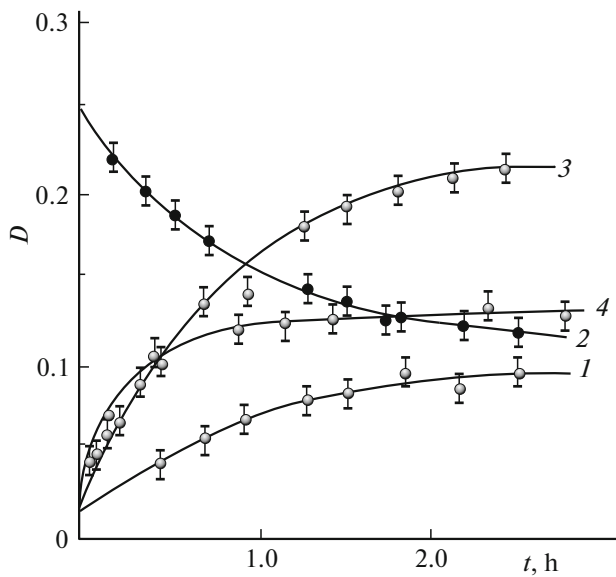


Fig. 3. Variation in the optical density of the IR absorption of polypropylene (PP) subjected to the electric-discharge effect depending on the treatment time: (1) 3200 cm^{-1} —OH, (2) 4327 cm^{-1} —CH₂, (3) 1720 cm^{-1} —C=O, and (4) 1278 cm^{-1} —C—O—C groups. The thickness of the air inclusion, where the electric-discharge appears, $d = 4\text{ mm}$. The voltage applied to the plasmatron was $U = 20\text{ kV}$.

ation in the maximum of the dependence of U_{epd} on the PbTiO_3 concentration for preliminarily plasma-crystallized composites can be attributed to an increase in the electronegativity of PP molecules because the electronegativity of macromolecules of the polymer matrix, as was already mentioned, strengthen local electric fields. A noticeable increase in δ and ϵ after plasma crystallization can also be associated with increasing electronegativity of macromolecules of the polymer matrix. Thus, our numerous experimental results show that the electret effect in polymer—ferroelectric piezoceramics composites is determined by several parameters of the piezophase, i.e., is a multifactor function. Therefore, optimization of the fabrication technology of composites is a topical problem when developing multifunctional active composites—electrets, ferroelectrics, and pyroelectrics. It is necessary to develop a mathematical model of the mentioned composites, based on which, separate characteristics of the polymer—ferroelectric piezoceramics system should be optimized [26–28]. The numerous experimental results obtained by us show that the variation in interphase interactions is affected not only by the electronegativity of the polymer phase performed by plasma crystallization but the electronegativity of cations of the piezophase as well. Therefore, we in parallel investigated the influence of the electronegativity of cations of the piezophase on the piezoelectric and insulating properties and electret

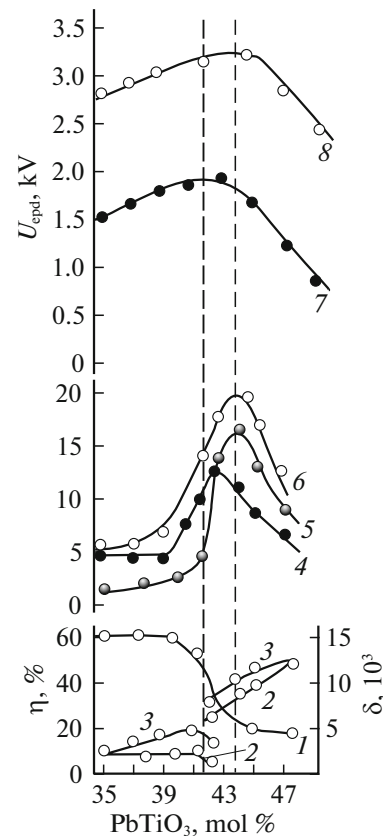


Fig. 4. Dependences of (1) η and (2) δ (2) before and (3) after polarization, ϵ/ϵ_0 ((4)—nonpolarized), and $\epsilon_{33}^T/\epsilon_0$ ((5) polarized) on the PbTiO_3 content in it, i.e., in the TsTS-21 system; of ϵ_k^T/ϵ_0 ((6) polarized at T_{pd} higher than the Curie temperature $T_c = 453\text{ K}$) of the piezophase on the PbTiO_3 content in it, i.e., in the PbTiO_3 — PbZrO_3 system; (7) dependence of U_{epd} of the PP + 5 vol % TsTS-21 composite on the PbTiO_3 content in the PbTiO_3 — PbZrO_3 piezophase; and (8) dependence of U_{epd} of the PP + 5 vol % TsTS-21 composite on the content of the PbTiO_3 composite of the PbTiO_3 — PbZrO_3 piezophase for the preliminarily plasma-crystallized PP + 5 vol % TsTS-21 samples. The piezoparticle size is $150\text{ }\mu\text{m}$, $E_{\text{pol}} = 2\text{ MV/m}$, $T_{\text{pol}} = 413\text{ K}$, and $t_{\text{pol}} = 0.5\text{ h}$. Composite PP + 5 vol % TsTS-21.

charge of the composites. Figure 5 shows the parameters of the composites that depend on the electronegativity of the elements B^{IV}. The degree of covalence B^{IV}—O increases with increasing electronegativity. It is seen that parameters (d_{33} , Q_r , and ϵ_{33}) decrease steadily with increasing electronegativity B^{IV}. It was already noted above that this fact is determined by the resistance of local energy levels of the quasi-forbidden band of the polymer phase to the effect of mechanical stresses during polarization, i.e., by the activation energy of the neutralization of polymer traps by mechanical perturbation. Along with the concentra-

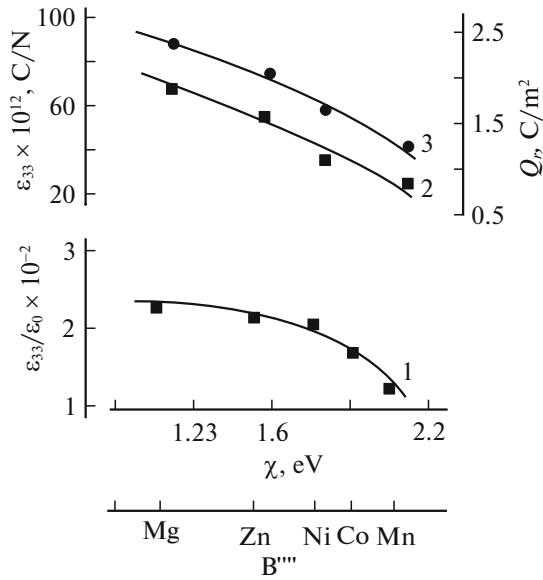


Fig. 5. Measurement of the parameters of the HDPE-based composite depending on the electronegativity χ of cations— B''' of the $PbTiO_3$ — $PbZrO_3$ — $PbB'_{1-\alpha}B''_{\alpha}O_3$ — $PbB'''_{1-\beta}B''_{\beta}O_3$. (1) ϵ_{33}/ϵ_0 is the composite permittivity, ϵ_0 is the electric constant of 8.854×10^{-12} F/m; (2) composite electret charge Q_r ; and (3) composite piezo module d_{33} . $E_{pol} = 3.5$ MV/m, $T_{pol} = 393$ K, and $t = 0.5$ h.

tion of local levels in the quasi-forbidden band of the polymer phase, it is necessary to have a large energy burial depth of the traps. In this context, we investigated the spectra of the thermally stimulated depolarization current of polarized piezoelectric composites. The spectra of the thermally stimulated depolarization current allow us to acquire information on the charge state of the phase interface and the stability of the boundary charge Q_r , depending on the electronegativity of cations of the piezophase of the composite. The spectra of the thermally stimulated depolarization current for HDPE and HDPE— $PbTiO_3$ — $PbZrO_3$ — $PbB'_{1-\alpha}B''_{\alpha}O_3$ — $PbB'''_{1-\beta}B''_{\beta}O_3$ composites are presented in Fig. 6. The composites differ in terms of the electronegativity of cations of the piezophase: Mn (2.12 eV), Co (1.84 eV), Ni (1.56 eV), Zn (1.32 eV), and Mg (1.23 eV) or the degree of cation—oxygen covalence (B''' —O), i.e., Mn—O, Co—O, Ni—O, Zn—O, and Mg—O. We assume that the interaction between polymer chains and the piezophase surface noticeably decreases with an increase in the degree of covalence of the B''' —O bond, i.e., interphase interactions decrease. This is in turn accompanied by an increase in the mobility of polymer chains in the vicinity of the surface of piezoceramic particles, the activation energy of charges accumulated at the phase interface decreases, and the region of the polymer phase, the structure of which is formed under the influence of the

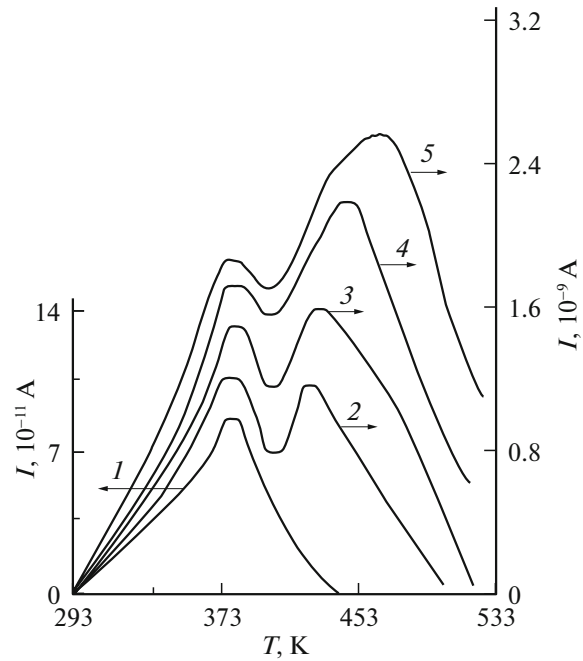


Fig. 6. Spectra of the thermally stimulated depolarization current of HDPE and composites: HDPE— $PbTiO_3$ — $PbNb_{2/3}Mg_{1/3}O_3$ — $PbNb_{2/3}B'''O_3$. (1) The HDPE spectrum, (2) the spectrum of the composite based on the piezophase with a Mn cation, (3) the spectrum for the composite based on the piezophase with a Ni cation, (4) the spectrum for the composite based on the piezophase with a Zn cation, and (5) the spectrum for the composite based on the piezophase with a Mg cation. Polarization conditions of the composites: $E_{pol} = 4.5$ MV/m, $T_{pol} = 393$ K, $t_{pol} = 0.5$ h, and the volume content of the piezo phase $\Phi = 50$ vol %.

surface of piezoelectric particles, is limited. These factors are very well reflected in the spectra of thermally stimulated depolarization current (Fig. 6). Based on these spectra, we can conclude that the use of ferroelectric piezoceramics with cations of low electronegativity and B''' —O covalence as a piezophase leads to the enhancement of adsorption interactions between the polymer phase and piezophase and the displacement of the second spectral peak towards high temperatures. The concentration of charge localization centers at the phase interface rises, the mobility of macromolecular chains of the polymer phase decreases, the activation energy of electric charges stabilized during polarization at the phase interface increases and, consequently, the local field at piezoelectric particles increases.

The above-mentioned allows us to construct in the first approximation a possible model of electret and piezoelectric composites microstructured by the piezoelectric particle (Fig. 7). To reveal the formation mechanism of the electret and piezoelectric effects, it is necessary to analyze the occurring physical models with the purpose of their improvement [26–28], here-

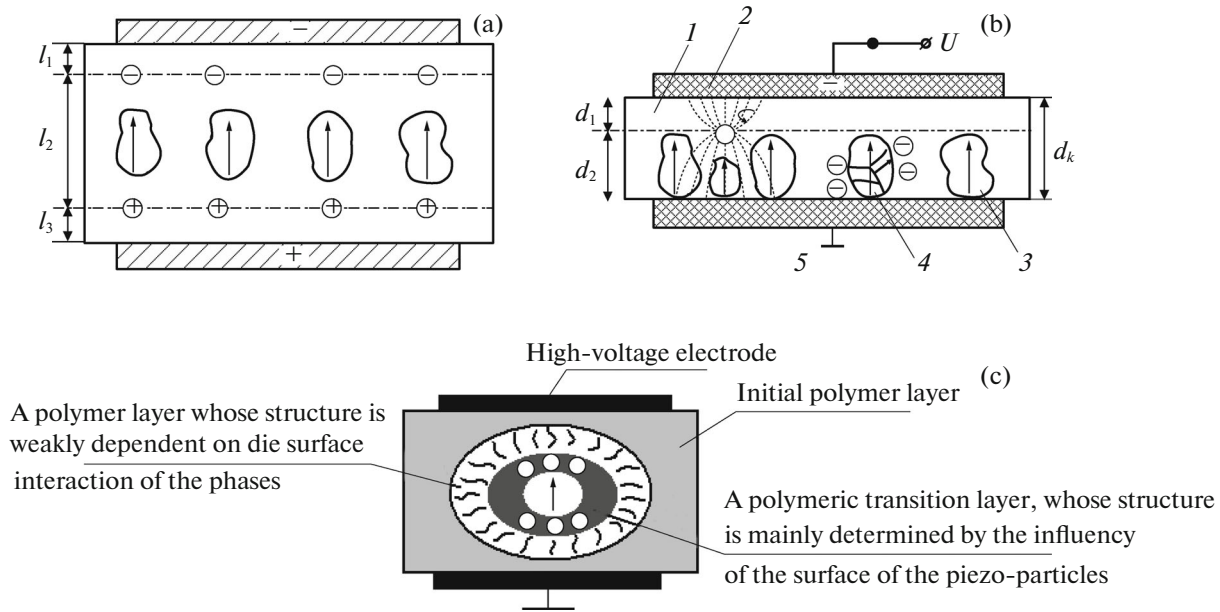


Fig. 7. Polarization model of the piezoelectric particle in the electret composite: (a) l_1 and l_3 are the thicknesses of regions in direct contact with the electrode and l_2 is the region in direct contact with a piezoparticle; (b) (1) the polymer layer in direct contact with the electrode, (2) the injecting electrode, (3) the piezoceramic particle, (4) the piezoelectric particle with domains that had no time to orient along the polarization field (inefficient quasi-neutral system), and (5) the grounded electrode; and (c) model of the interphase layer in the polymer–ferroelectric piezoceramic system.

with taking into account the role of electronegativity of the composite components. It was already mentioned that the electronegativity of cations of the piezophase and polymer matrix limits the mobility of macromolecules, which in turn requires the formation of a thinner interphase polymer layer with a high-quality crystal structure (Fig. 6). Such a structure of the composite and boundary layer promotes more intense charge accumulation at the boundary of the piezophase and polymer.

Figure 7 proposes a possible pattern for a model of the composite material, which allows us to explain in the first approximation the formation of the above-mentioned effects with electron–ion and polarization processes taken into account (Figs. 7a–7c). Figure 7a shows the stylized formation pattern of an oriented domain–injected charge quasi-neutral system [16], which allows us to explain the mechanism of development of the initial stage of the electret and piezoelectric effects. Figure 7b shows possible electron–ion and polarization phenomena in the polymer–ferroelectric piezoceramic composite during thermal electretting. Initially electrons are injected into the composite under conditions of the simultaneous effect of electric field (E_e) and temperature (T_e). Let us assume that the charge is stabilized in the composite bulk at a distance of d_1 from the high-voltage electrode surface. It is seen that the injected charge is stabilized in the quasi-forbidden band of the polymer phase and forms a local field leading to domain orientation according to the

strength vector of the polarization electric field. This field in particular promotes the efficient polarization of the piezophase (Fig. 7b). The probability of the formation of a quasi-neutral system consisting of migrating (injected) charges and domains that had no time to orient along the polarization field is more interesting from the viewpoint of efficient composite polarization in general. This means that this type of quasi-neutral system is inefficient and does not participate in the formation of electret and piezoelectric effects. This problem has tremendous value in developing composites of various applications and optimizing their parameters [26–28]. Here, we should take into account the conductivity of the polymer matrix and mobility of domains of the piezoelectric phase of the composite materials. It is known that the conductivity of dielectrics, including heterogeneous dielectric systems, is determined in the first approximation as $\sigma = \mu Ne$, where μ is the mobility, N is the charge-carrier concentration, and e is the elementary charge. For our problem, it is necessary to take into account the mobility and magnitude of the local mechanical stress of piezophase domains. When determining the electret charge (the electret potential difference), it was noted that it substantially depends on the structure of the piezophase of the composites: rhombohedral, tetragonal, and heterogeneous (Fig. 2) [4]. It is known that the piezoceramic samples with a rhombohedral structure have larger domains (5–20 μm) compared with ceramics with a tetragonal structure (5–10 μm) [4]. It is also known that the domain mobility of the piezoc-

eramic samples with a tetragonal structure (mainly 90-degree) is smaller than a similar parameter for piezoceramic samples with a rhombohedral structure with 109- and 71-degree domains. Therefore, we should take into account the correspondence of the mobility of the piezophase domains with the mobility of injected electrons in the polymer phase during electrothermal polarization. The mobility of electrons and ions in a polymer phase, which formed under the influence of the piezoparticle surface, i.e., in the transient layer, is of special value (Fig. 7c). Here we note that determination of the interrelation between the relaxation times of domains in the piezophase and injected charges in the transient layer require more careful investigation, which is beyond the scope of our study. In our opinion, it is more optimal to accept a transient layer with a crystal structure. In this case, we can expect the formation of an efficient quasi-neutral system larger in terms of concentration but with a lower efficiency.

4. CONCLUSIONS

The following resulted from our study:

(i) the electronegativity of the piezophase and polymer matrix play a decisive role in the formation of a high electret state in the composites;

(ii) the mobility of the piezophase domains and injected electrons in the polymer matrix substantially affects the formation of the efficient quasi-neutral system in polymer–ferroelectric piezoceramic composites; and

(iii) an improved model explaining the formation mechanism of the electret effect is proposed.

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