PHYSICS OF COMPOSITE ACTIVE MATERIALS

KURBANOV M.A., SHAKHTAKHTINSKY M.G., MUSAEVA S.N.

Institute of Physics Azerbaijan National Academy of Sciences

On the basis of analysis of theoretical and experimental investigations of properties of active composites polymer-piezoelectric it has been shown, that interaction between the phases plays a significant role in formation of the piezo-, piroelectric and posistor effects in these materials. Specificity of physics of the compositional structures must be sequential accounting of different kind of interactions on a phase interface. For example, the effects connected with the electrothermopolarization of a piezophase in the polymer-piezoelectric system are explained on the basis of model, demonstrating the creation of the quasineutral system oriented domain and a charge stabilized on the phase interface in the polymer. Such an approach allows accordingly to explain the piezo-, piro-, photoelectric, piezoresistive, varistor and electret effects in the composites and their electro- and thermophysical properties.

I. Introduction

The composites are widely applied almost in all fields of technology and constitute the background of the most natural materials. In this connection a lot of works are devoted to their fabrication technology and studying of their microscopic characteristics [1,2], significantly, of the practical directionality. As far as the structures and feature of composites are extremely various, the general approach for explanation of their properties the formation practically is absent.

The most common representation of composites feature formation is based on account of characteristics of the constituents. In this case the geometry (size, shape) and their initial position are taken into account. The interaction between the phases is considered negligible, i.e. integral characteristics of composites are taken additive. By this sort of approach there is no necessity in separation of composite material physics. It follows also from adopted in literature definition of composites [3,4] as the heterogeneous systems, obtained from two or more components with conservation of individual properties each of them. However, one cannot neglect the interaction between the phases formed the composites as far as it becomes decisive in formation of their properties for many cases. The obvious example of the impossibility of determination of the integral features of composites starting from addictively of the contributions of the phases, is a system consisted of sequentially connected phases, between which *p-n* junction is formed. If one calculate the conductivity of such system by addition of the separate phase conductivities, then the absurd result will be obtained, because a system conductivity as a whole is determined by heterojunction.

Below we shall make an attempt to show the necessity of accounting of the Interface interaction, the electron states of phase interface and charge stabilizations in it in formation of the composite properties on example of disordered polymer-piezoelectric system.

II. Experiment

High-density and low-density polyethylene (HDPE and LDPE), polypropylene (PP) and polyvinylidene fluoride (PVDF) are used as a matrix of composites. Multicomponent piezoceramics of lead-zirconate-titanate family of PCR - piezoceramics Rostovskie type are used as an active filler. In this paper foreign analogues of the used piezoceramics PCR are presented. Samples are obtained based on the homogeneous mixture polymer-piezoparticles by hot pressing method. Diameter of the piezoparticles was changed from 63 to 100 μm. Thickness of samples was varied from 80 to 200 μm . Piezocoefficient (d_{33}) of composites was measured in the quasistatic condition with the error not more 8%. Volume fraction of piezophase was changed from 40 to 70 %. Polarization of composites was brought off in simultaneously action of constant electric field and temperature. Magnitudes of electric intensity (E_p) and temperature (T_p) of polarization were limited by electric strength and melting temperature of samples.

III. Results and discussion

Let's consider the role of polarization processes taking place in a polymer matrix and on a polymer-piezoelectric interfaces on the basis of following experimentally stated facts:

1. The sophisticated dependence of piezocoefficient of composites vs electric fields E_p, polarization temperature T_p (fig 1 a, b).With increasing of E_p or T_P the composite piezocoefficient (d_{33}) is increased, passes via maximum and then is decreased. As far as the temperatures and electric field intensities of polarization are smaller than Curie temperature of piezophase and composites electric breakdown intensity correspondingly, then these dependencies mustn't have a maximum.

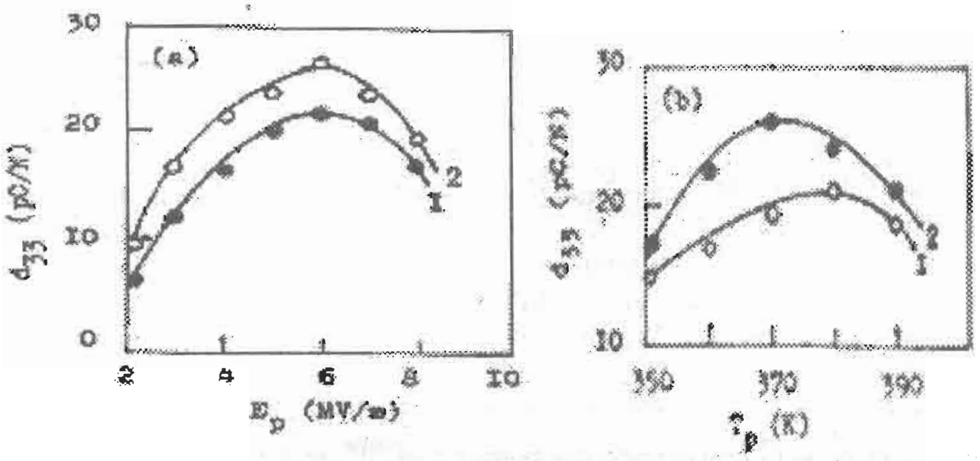

Fig. 1. Piezocoefficient d_{33} of PP/PZT -19 composite vs (a) electric intensity E_p at (1) T_p=373 K, (2) T_p =393 K and (b) temperature T_p of polarization at (1) E_p =4 MV/m, (2) E_p =6 MV/m. Volume fraction of piezoceramic is Φ = 0,5. Diameter of piezoparticles is 63 μm.

2. A comparison of d_{33} values of the composites on the base of the low and high-density polyethylene and polypropylene with the same piezofiller (fig.2) indicates that their piezoelectric coefficient corresponding to the optimum polarization conditions are significantly distinguished. A theoretical consideration of two phase system based on a polymer dielectric dispersed by piezoelectric particles uniformly distributed in volume, gives [1]

$$d_{33} = \Phi \cdot \frac{3\varepsilon_1}{2\varepsilon_1 + \varepsilon_2 + \Phi(\varepsilon_1 - \varepsilon_2)} \cdot \frac{5C_2 \cdot {}^2d_{33}}{3C_1 + 2C_2 - 3\Phi(C_1 - C_2)} , \qquad (1)$$

where ε_1 and ε_2 are dielectric constants of the polymer matrix (phase 1) and piezoceramics (phase 2), accordingly, C_1 and C_2 are appropriate elasticity modulus. Φ is the volume fraction of piezofiller, ${}^2d_{33}$ is a piezocoefficient of piezofiller.

With taking into account $\varepsilon_1 >> \varepsilon_2$ and $C_2 << C_1$ one obtains

$$d_{33} = \frac{15\Phi}{(1-\Phi)(2+3\Phi)} \cdot \frac{d_{33}}{\varepsilon_2} \varepsilon_1 \qquad (2)$$

It is seen, that the piezocoefficient of composite is proportional to ε_1 of polymer phase and piezoceramic's sensitivity ($^2d_{33}/\varepsilon_2$). Thus this experimental fact has no explanation within the scope of theory, since ε for the polymers of the pclyolefin chain are the same.

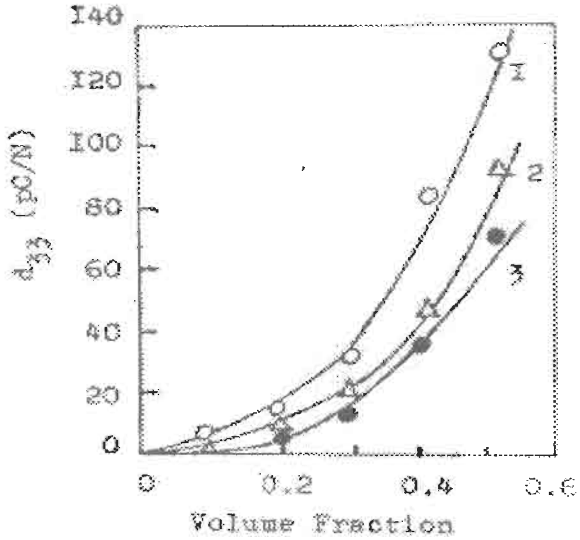

Fig. 2. Piezocoefficient d_{33} as a function of piezoceramic volume fraction of composites on the basic of polyolefms and piezoceramic PCR-3M. (1) PP/PCR-3M; (2) LDPE/PCR-3M; (3) HDPE/PCR-3M.

3. The comparison of the piezoelectric properties of the composites based on the same polymer with different piezoelectric fillers also shows that the piezocoefficients of the composites (fig. 3) are not proportional to the ratio $^2d_{33}/\varepsilon_2$, as it is predicted by theory.

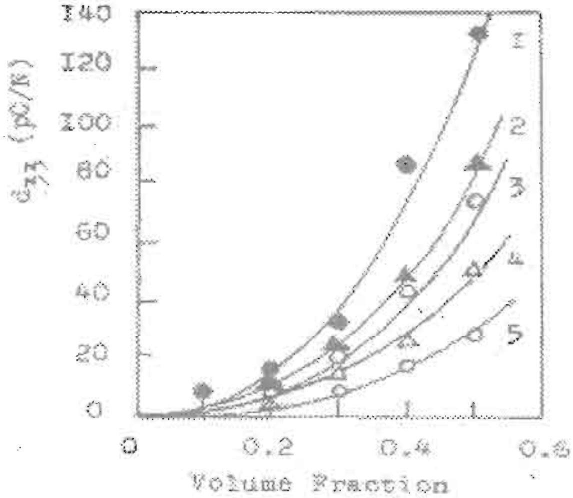

Fig. 3. Piezocoefficient d_{33} as a function of piezoceramic volume fraction for composites on the basic of polyethylene and piezoceramics with different d_{33}/ε_2%: (1) PP/PCR-3M, d_{33}/ε_2=0,24 for PCR-3M; (4) PP/PCR-1, d_{33}/ε_2=0,038 for PCR-1; (2) PP/PCR-5, d_{33}/ε_2=0,029 for PCR-5; (5) PP/PZT-19, d_{33}/ε_2=0,024 for PZT-19; (3) PP/PCR-7M, d_{33}/ε_2=0,0158 for PCR-7M;

4. It is seen from preceding results, that for the optimal polarization the composites have significant piezocoefficient, that points to high polarization of piezophase. Nevertheless the

effective field E acted on isolated spherical piezoparticle in composite is defined according to Frehlikh theory with taking into account $\varepsilon_1 \langle\langle \varepsilon_2$ by relatior

$$E = \frac{3(1+2\Phi)}{1-\Phi} \cdot \frac{\varepsilon_1}{\varepsilon_2} \cdot E_p \tag{3}$$

It is seen, that the field intensity per of piezoparticle during thermopolarization much smaller than the applied field of polarization E_p and coercive field of piezoceramic and consequently the piezoparticles must not be effectly polarized.

5. The attempts were made to explain the significant distinctions between the experimental data and theoretical calculations with taking into account a third phase supposedly arising as a result of the interaction of the piezoparticles and polymer [5].

However, the use of such model leads to nonreasonable values for parameters of the intermediate third phase, considered as fitted one for the agreement of the theory and experiments.

Numerous experimental results [6,8] for the composites based on pieroceramics and polymers with the various structures and polarities verify the facts listed above and point to necessity for different approach in explanation of the piezoelectric properties formation of composites.

In particular, all these facts may be explained if one takes into account an interconnection of charge accumulation processes in polymer matrix and piezophase polarization of composite.

A charge state of the composites has been investigated by the thermal stimulated depolarization method (TSD). In Fig.4 the typical TSD spectra of composites are shown.

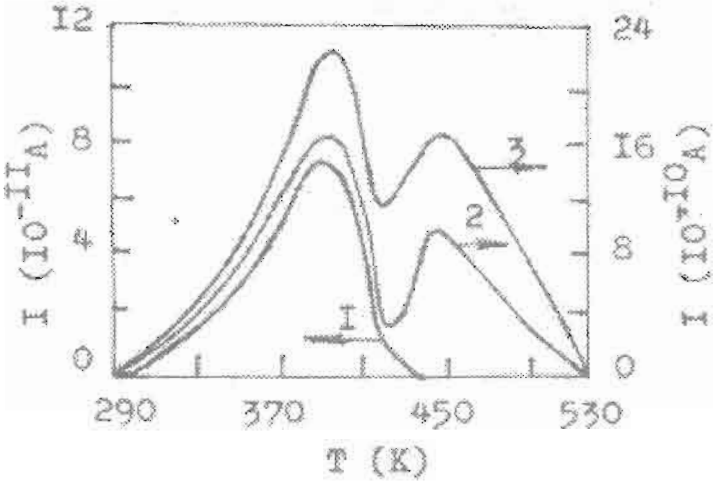

Fig. 4. Spectra of currents of thermal stimulated depolarization. (1) HDPE; (2) HDPE/PZT-19, Φ=0,3; (3) HDPE/PZT-19, Φ=0,4. Conditions of polarization: E_p=1,5 MV/m, T_p=373 K, time of polarization - 1 hour.

There are two maximums on spectra. The first, low temperature one is coincides with the TSD maximum of pure polymer. An activation energy of the first TSD maximum of the composite is close in a magnitude to the activation energy of TSD maximum of a base polymer and practically not depends on type of filler.

One can come to conclusion that the first maximum of TSD spectrum is caused by charge carriers liberation from the traps in polymer. The second maximum of TSD current of a composite in connected with a relaxation of charge captured by traps with significantly greater activation energy. One can support that these traps are located in boundary layer of polymer near the particles of the piezofiller.

A role of the charge stabilized on the polymer-piezoceramic interface in formation of the piezoelectric properties of the composites in verified by following experimental facts:

1. The dependencies of the total charge Q and charges Q_1 and Q_2 corresponding to the first and second TSD maxima vs E_p, T_p (fig. 5) and content of the piezofiller (fig. 6) have the same form as the dependencies d_{33} vs these parameters (fig. 1, 2, 3).

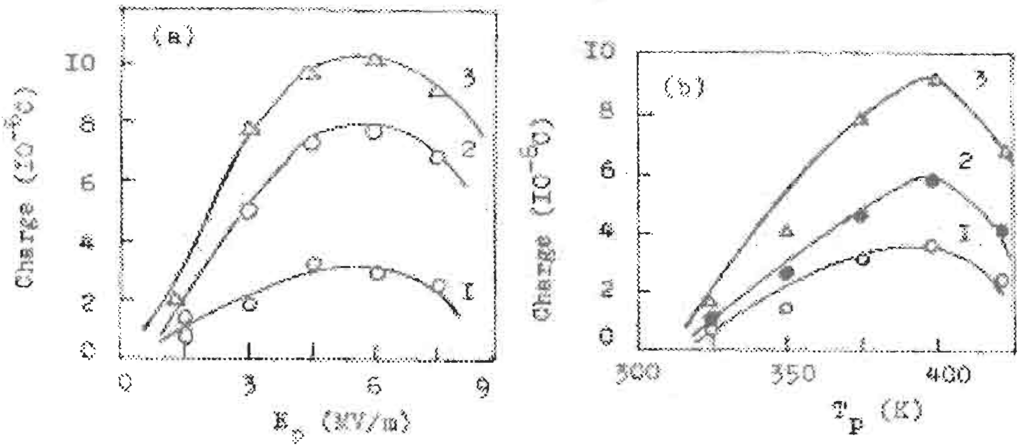

Fig. 5. Total charge Q(1) and charges corresponding to the first Q_1 (2) and the second Q_2 (3) maximuma of TSD current vs (a) electric intensity E_p and (b) temperature T_p of polarization for PP/PZT-19 composite.

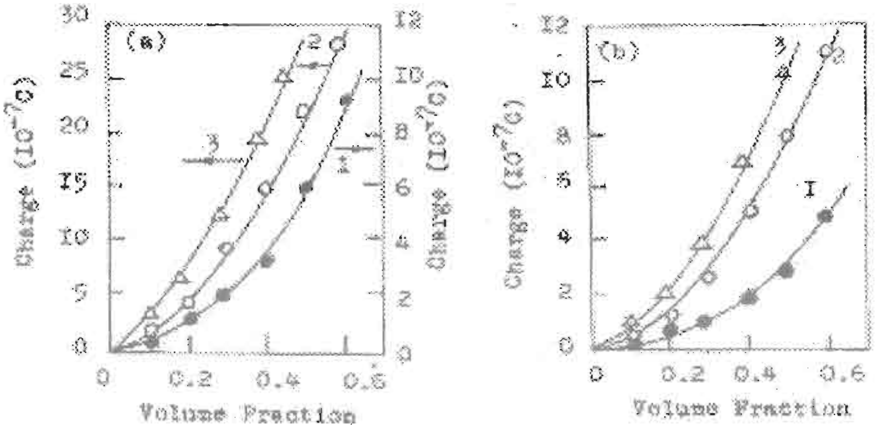

Fig. 6. (a) Total charge Q and (b) charge corresponding to the second maximum Q_2 of TSD current as a function of piezoceramic volume fraction. (1) HDPE/PZT-19; (2) PP/PZT-19; (3) LDPE/PZT-19; (4) HDPE/PZT-19 after heat

2. A direct proportionality a observed between the charge Q_2 and piezocoefficient d_{33} with a variation of Φ (E_p= const, T_p= const) (fig. 7), E_p (T_p= const) and T_p (E_p=const) (fig. 8). Such dependencies is not observed for the charges Q, Q_1 and d_{33} (fig.7,8).

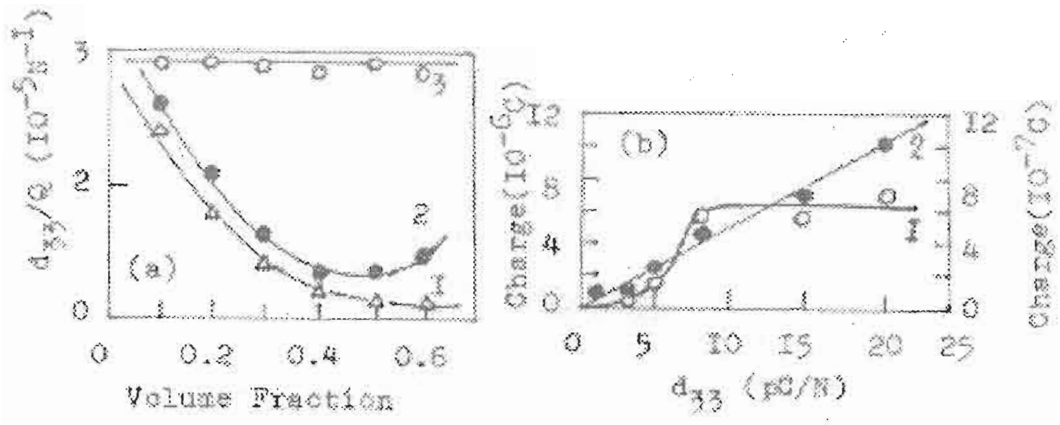

Fig. 7. (a) d_{33}/Q (1), d_{33}/Q_1 (2) and d_{33}/Q_2 (3) as a function of piezoceramic volume fraction; (b) Q_1(1) and Q_2 (2) as a function of piezocoefficient for HDPE/PZT-19 .

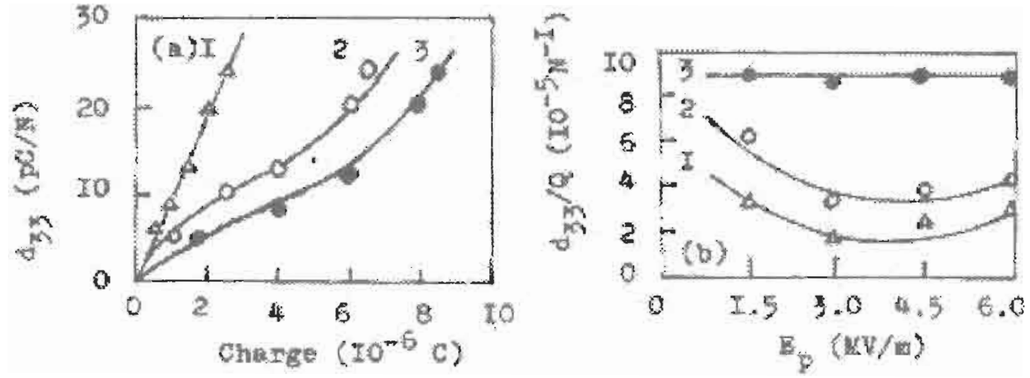

Fig. 8. Piezocoefficient as a function of charges Q_2 (1), Q_1 (2) and Q (3) obtained at variation of E_p and T_p =373 K. (b) d_{33}/Q (1), d_{33}/Q_1 (2) and d_{33}/Q_2 (3) as a function of E_p for PP/PZT-19 , Φ= 0,5.

3. The values of Q_2/Q_2 and d_{33}/d'_{33} practically is constant (fig.9), but Q/Q' and Q_1/Q'_1 entirely relaxed (fig.9 and 10) with the heating till temperature of TSD spectrum minimum.

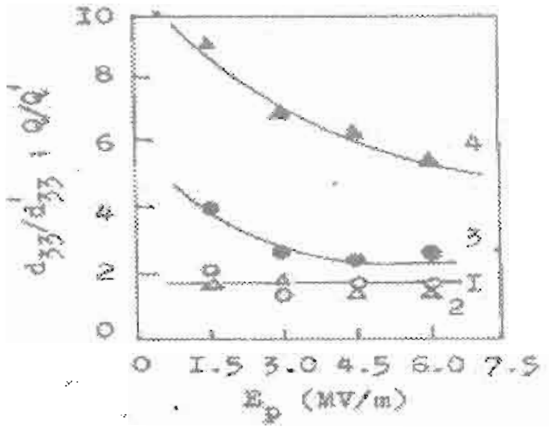

Fig. 9. d_{33}/d_{33} (o), Q_2/Q'_2 (Δ), Q_1/Q'_1 (Δ) and Q/Q' (o) E_p for PP/PZT-19 , Φ=0,5; T_p =393 K, Q and Q' are total charges before and after heat treatment, correspondingly; Q_1 and Q'_1 are charges of the first maximum of TSD currents before and after heat treatment, correspondingly; Q_2 and Q'_2 are charges of the second maximum of TSD currents before and after heat treatment, correspondingly; d_{33} - piezocoefficient after heat treatment.

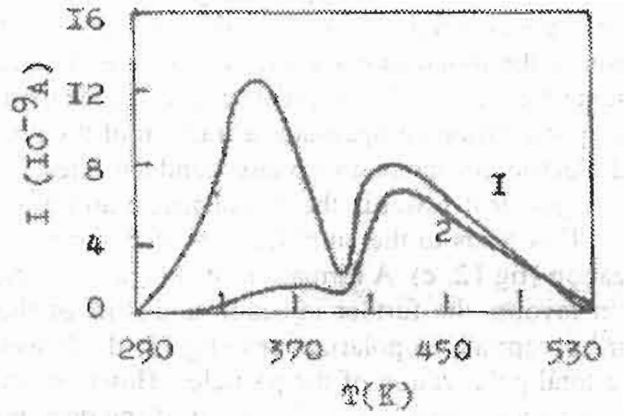

Fig. 10. Curves of thermal stimulated depolarization (TSD) of LDPE/PZT-19. Curve 1 - at the first heating up to 413 K. Curve 2 - at the second heating (after heat treatment). E_p =1,5 MV/m, T_p =373 K, Φ=0,5.

4. The simultaneous relaxation Q_2 and d_{33} is observed at temperatures beyond second TSD maximum temperature (Fig. 11). So that above listed experimental results indicate that the tight binding between the accumulated charges on the phase interface in polymer matrix and piezophase polarization is formed. Such interconnection may be explained on the basis of a simplified model showed in fig. 12.

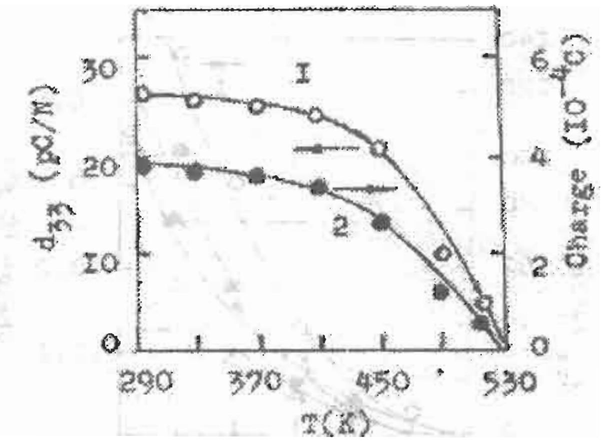

Fig.1 1. Q_2 and d_{33} vs temperature for LDPE/PZT-19 composite.

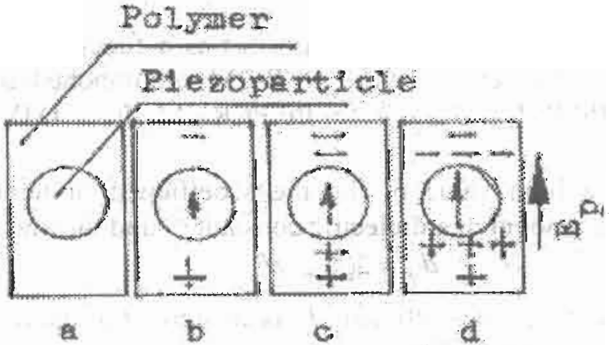

Fig. 12. Model of flowing of injected charges to polymer- piezoparticle and polarization of piezophase in the field of injected charges. E_p - polarization intensity.

According to this model the polarization process goes on by following way. At $E_p=0$ a total polarization of piezoparticles is equal to zero (fig. 12,a). During electrothermopolarization in the initial moment the charges are injected from electrod to the composite and at the same time insignificant polarization of piezoparticles takes place (fig. 12, b). This insignificant polarization compensates a fraction of the per particle external field in accordance to the dielectric constants and phase conductivities for choosed T_p and E_p. Later on the injected charges drift towards the piezoparticle and are localized on the traps near the phase interface. This leads to the amplification of the local field on a piezoparticle and increase its polarization (fig.12, c). A formation of the quasi-neutral complexes injected charges-oriented domain favours the further injection and drift of the charges towards the piezoparticles and their appropriate to polarizations (fig.12, d). It would seem that process must be continued till a total polarization of the particles. However, in practice a number of facts may disturb it, for instance a deficiency of amount of the deep traps on the interface, a screening of the piezoparticles by accumulated charges and etc.

The efficiency of the composites becomes dependent on the piezoceramic structure. In fig.13 for example, the dependencies of d_{33} and Q on a volume fraction of piezophase of various structure are shown. It is seen, that the magnitude of stabilized charge and the piezocoefficient value are greater in the case of using of the piezoceramic of rhombohedral (PCR-3M) structure as a piezofiller compared with the tetragonal (7) structure (PCR-7M) piezoceramic. At the same time the base ceramics have reverse relation of the piezocoefficient.

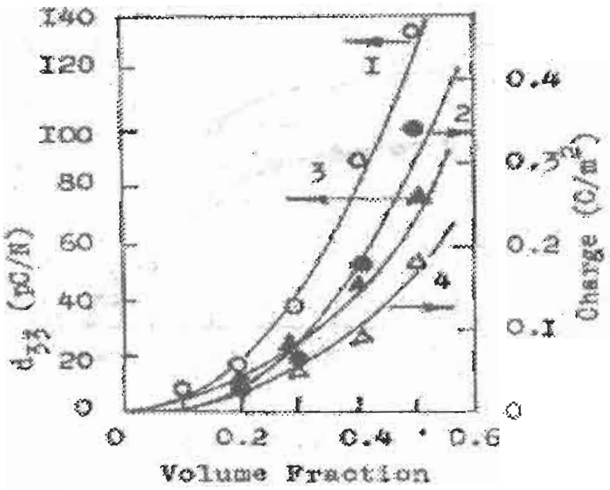

Fig.13. d_{33} (1, 3) and Q_2(2, 4) of composites as a function of piezoceramic volume fraction of different structure. PCR-3M - rhombohedral; PCR-7M - tetragonal. (1, 2) for PP/PCR-3M; (3, 4) for PP/PCR-7M . $\mathbf{E_p}$ = 3MV/m, $\mathbf{T_p}$ = 410 K.

In literature [9] a high value of the piezocoefficient of tetragonal piezoceramics is associated with a large amount of a dielectric constant ε and the known relation is used:

$$d_{ij} = 2Q_{12}\varepsilon_0\varepsilon P_r \tag{4}$$

where Q_{12} is the electrostriction coefficient, P_r is an amount of the reorientational polarization of domains different from those of 180°.

Dielectric constant in composites is significantly smaller than that of in the piezoceramics because of a low value of ε at a polymer phase. Therefore a main role plays P_{r1} that is greater in the rhombohedral ceramics. On the other hand in the rhombohedral ceramics a mobility of the domain walls is greater, that in turn leads to increasing of a charge moving to the interphase boundaries. This is a cause of a great magnitude of Q_2.

Thus the above model accordingly explains both the polarization curves and the dependencies $d_{33} = f\ (E_p, T_p)$, and dependence of the piezocoefficient on the piezofiller structure. An additional verification of a workability of the proposed model one may obtain from calculation of d_{ij} of the composites using the expression (4). Taking into account a major role of the interface charges in piezoparticles polarization it should be used Q_2 instead of P_T in formula (4). A magnitude of Q_{12} for composites is taken to be equal to its value for a base piezoceramic. Such approach is justified by the fact, that the polarization is concentrated in the piezophase.. Thus the experimental data and suggested model demonstrate a significant role of one kind of the interface interaction in the formation of the composite properties. This interaction leads to seemed anomalous dependencies of composite properties on a composition. In fig. 14 as example the dependence of d_{ij} on Φ for a composite PVDF/PCR-3M is shown. It is seen, that the amount of the composite piezocoefficients for the certain compositions becomes greater than d_{ij} of both components.

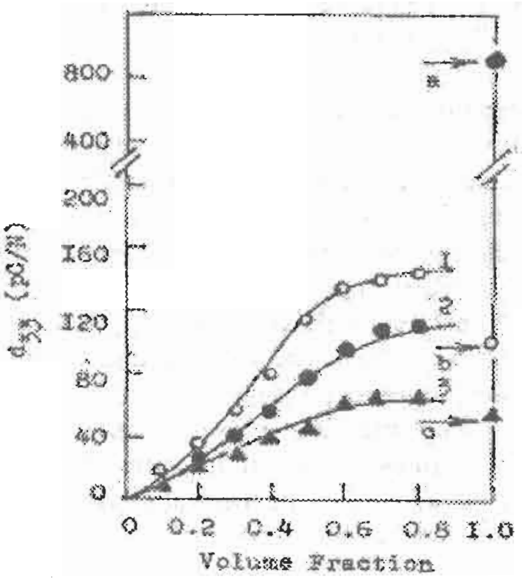

Fig. 14. Variation of piezocoefficient of composites with piezoceramic volume fraction of different structure. (1) PVDF/PCR-3M; (2) PVDF/PCR-7M; (3) -PVDF/PCR-37, a (•), b (o) and c (Δ) points correspond to the value of piezocoefficient of starting ceramics of PCR-7M, PCR-3M and PCR-37. $E_p = 3, 5$ MV/m, $T_p = 393$ K, time of polarization - 1 hour.

If the interface interactions would not play a significant role in the formation of piezoelectric effect, then such maximum must be absent. In the case, when the interaction between the charges localized in a polymer phase and particles polarization as taking into account, this maximum points to the best polarization of piezophase in a composite compared to a base ceramic. In general case the appearence of maxima on diagrams composition-composite properties shows the significant role of the interaction on the interface. In particular, the above pictured interaction on the interface, that is lead to the formation of the system of oriented domain-localized boundary charge, explains the piezoelectric and electret effects in composites [10-12]. In fig. 15 a piroelectric current curve for a composite PVDF/PCR-3M is shown.

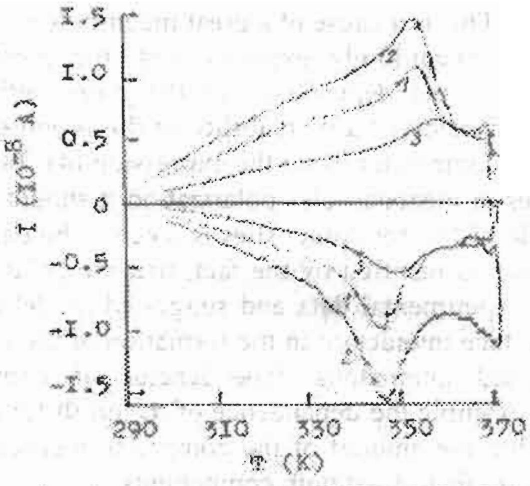

Fig. 15. Curves of the piroelectric current for PVDF/PCR-3M. Φ=0,4, t_p= 0,5 hour.
(1) E_p=3 MV/m, T_p=353 K; (2) E_p=3 MV/m, T_p=373 K;
(3) E_p=3 MV/m, T_p = 413 K.

It should be noted, that the piroelectric effect in composites becomes greater than in base components. This is in agreement with the above pictured model, which explains an occurrence of the large reorientational polarization responsible for piezoelectric effect [9-11]. With the existance of coupling charge-domain system, the occurrence of the stable electret state in the polymer-piezoelectric composites with a fitted composition is also explained.

A stabilization of the boundary charges on the phase interface and their relaxation significantly depends on a structure of polymer on the phase interface. In its turn this structure is defined by interaction between the polymer chains and piezoparticle surface. In particular, in fig.16 the temperature dependencies of the piezocoefficient and thermal expansion coefficient *(α)* of composites based on the single matrix and piezoceramic of rhombohedral and tetragonal structures are shown. It is seen, that a temperature decline of the piezocoefficient and a noticeable increase of the thermal expansion coefficient is higher in the case of a piezofiller of the rhombohedral structure.

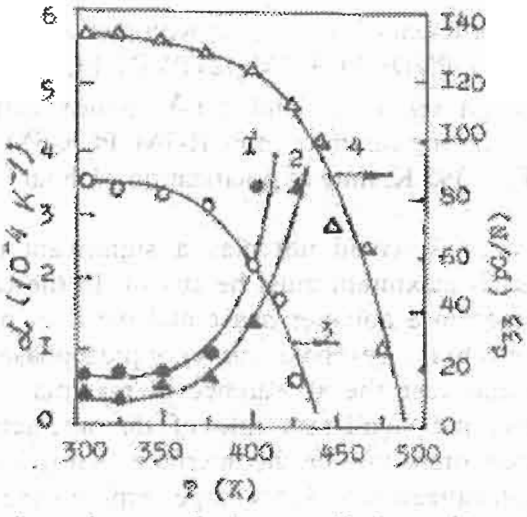

Fig. 16. Temperature dependences of piezocoefficient d_{33} and thermal expansion coefficient. LDPE/PCR-7M (1, 3) and LDPE/PCR-3M (2, 4) .

It should be noted, that the piezocoefficient decline is associated with the boundary charges relaxation, which in its turn is defined by mobility of the polymer chains. It shows that the interaction of the polymer chains with the piezoparticles surface of rhombohedral structure is stronger. By the same way the dependencies of a vs T are explained also.

A subsurface property was an effect on a posistor effect in a composite. It is known, that the posistor effect in the piezoceramics is associated with a transition from a ferroelectric phase to paraelectric one. In polymers this effect practically is absent. In the composites a significant posistor effect is observed (fig. 17).

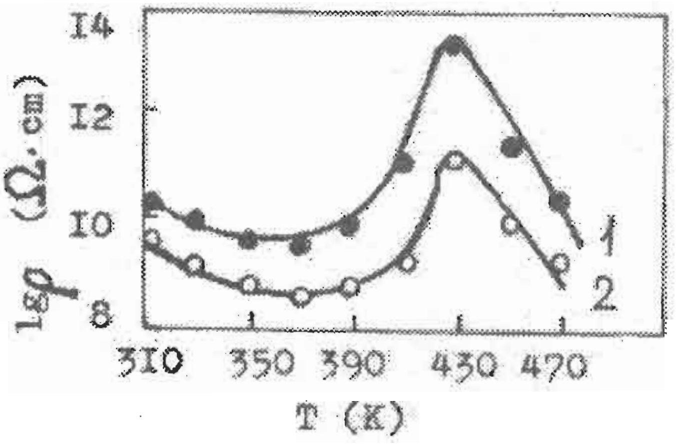

Fig. 17. Temperature dependence of volume resistivity logarithm lg p of PP/PCR-3M (curve 1) and PP/PZT-19 (curve 2) composites.

It can not be associated in composites with phase transition in piezoparticles, as far as a temperature of sharp increase of composite resistance is remarkably lower than the piezophase Curie temperature. The posistor effect in composites is associated with a sharp changing of the potential barrier on the phase interface in fusion of ordered phase of a polymer matrix [13,14]. Such effect is specific only for the composites and may be a basis of a new structure-sensitive method of their investigation. A decrease of mobility of the polymer chains because of their interaction with a piezoparticle surface leads to a change both the occurrence temperature and half-width of the posistor peak (fig. 17) when considering above the piezoelectric, piroelectric, electret and posistor effects in composites it was shown that their formations are determined by interaction between the phase on the interface. In this connection a definition of the compositional materials as heterogeneous systems with the keeping of phase individualities becomes nonrigorous. At least on the boundary layer there is occurred a significant change of the structure and microscopic parameters, such as for instance density, thermal expansion coefficient, thermal conductivity, heat capacity and etc. On the other hand a Coulomb interaction on the phase interface leads to a greater polarization of piezoparticles as a whole. There may be many kind of interactions. A contributions of different interactions naturally well be depend or considered properties. One of a principal problems of a compositional material physics must be location of major types of interactions on the phase interface and their proper allowance in formation of the corresponding effects.

In this paper such consideration is carried out only for one example of the composites. At the same time a similar consideration may be extend on the all compositional materials. Particularly, in our works [10-15] it is shown that the interaction on the phase interface plays a significant role in the formation of the photo-, piezoresistive, varistor and photovoltaic effects.

IV. Conclusions.

It is shown, that complex piezoelectric, electret and piroelectric properties in polymer-piezoelectric composites, their change on conditions of electrothermopolarization, an individual characteristics of phase and cubic content is consultationly explained by the formation of interconnected system polarized piezoparticle - localized on the phase boundary charge in polymer.

Interaction between the phases in the polymer-piezoelectric system leads to change of supermolecular structure of boundary layer which determines the formation of posistor, thermal and electric properties of composites.

Physics of composition structures must be based on consistent account of contributions of different type of interactions between the phases in the formation of specifical properties of composites.

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KOMPOZİT AKTİV MATERİALLAR FİZİKASI

QURBANOV M.Ə., ŞAHTAXTİNSKİ M.H., MUSAYEVA S.N.

Polimer-pyezoelektrik aktiv kompozitlərin xassələrinin nəzəri və eksperimental tədqiqi əsasında göstərilmişdir ki, bu materiallarda pyezo-, piroelektrik və pozistor effektlərinin formalaşmasında fazalar arasında qarşılıqlı təsir əhəmiyyətli rol oynayır. Kompozit quruluşlar fizikasının spesifik xüsusiyyəti fazalar sərhəddində müxtəlif növ qarşılıqlı təsirləri ardıjıl olaraq nəzərə almaqdan ibarətdir. Məsələn, polimer-pyezoelektrik sistemdə pyezofazanın elektrotermopolyarizasiyası ilə bağlı effektlər oriyentasiya olunmuş domen və polimerin sərhəddində stabilləşmiş yük kimi kvazineytral sistemin yaramasını göstərən model əsasında izah olunur. Bu jür yanaşma kompozitlərdə piroelektri, elektret. Fotoelektrik, pyezorezistiv və varistor effektlərinin və onların elektrofiziki və istilik xassələrini əlaqəli şəkildə izah etməyə imkan verir.

ФИЗИКА КОМПОЗИЦИОННЫХ АКТИВНЫХ МАТЕРИАЛОВ

КУРБАНОВ М.А., ШАХТАХТИНСКИЙ М.Г., МУСАЕВА С.Н.

На основании анализа теоретических представлений и экспериментальных исследований свойств активных композитов полимер-пьезоэлектрик показано, что взаимодействие между фазами играет существенную роль в формировании пьезо-, пироэлектрического и позисторного эффектов в этих материалах. Спецификой физики композиционнных структур должен являться последовательный учет различного вида взаимодействий на границе фаз. Например, эффекты, связанные с электротермополяризацией пьезофазы в системе полимер-пьезоэлектрик, объясняются на основании модели, демонстрирующей образование квазинейтральной системы – ориентированный домен и стабилизированный на границе раздела фаз в полимере заряд. Такой подход позволяет согласованно объяснить пироэлектрические, электретные, фотоэлектрические, пьезорезистивные и варисторные эффекты в композитах и их электро- и теплофизические свойства.