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THE MOLECULAR MOBILITY OF THE EPOXY BINDER IN A MODIFIED COMPOSITES BY ELECTRIC-HAMMER

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Summary. In this work, the study of molecular mobility in the surface layers by the method of determining the dielectric characteristics of materials modified by electrospark hydraulic shock, determined the optimal content of the dispersed filler. Comparative data of the tangent of the dielectric loss angle of treated and untreated composite materials are presented.

Key words: electric-hammer, molecular mobility, composite.

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Statement of the problem. Polymer composite materials (CM) on epoxy resins basis are widely used in technology to form coatings for various purposes. The main requirements for these products and coatings on their basis are the comprehensive increase in their molecular and mechanical characteristics, which leads to experimental studies of the dynamics of the processes of CM structure formation at different modes and specifically selected stages of polymerization of materials [1].

Analysis of available research. The study of physical and chemical processes in the polymer epoxy matrix during electrospark hydrotreatment (ESHT) has not yet been sufficiently studied [2]. In this regard, it is important to conduct research on the impact of ESHT on the structural characteristics and physical and mechanical properties of raw materials and modified materials.

Objectives of the research are to investigate the temperature characteristics of relaxation processes in the original and modified epoxy matrix, determine the temperature dependences of dielectric losses of epoxy composites in electrospark hydrotreated and untreated composites, as well as to investigate the temperature characteristics of relaxation processes in modified composites.

Formulation of the problem. Study of molecular mobility in the surface layers by determining the dielectric characteristics has shown that the presence of the phase separation boundary, due to the introduction of a dispersed filler into the oligomeric system, leads to significant change in the relaxation behavior of the polymer at the phase separation boundary [3, 4]. This also causes a change in the glass transition temperature and the expansion of the spectrum of relaxation times, which, in turn, indicates a change in both the conformational set of macromolecules and supramolecular formations in the surface layers. Herewith it should be noted that the effect of conformational changes on the properties of the surface layers is complemented by the energy interaction between the surface of the dispersed particles and the oligomeric binder [5]. This interaction, in turn, depends on the chemical and magnetic nature of the filler and determines the adhesive strength at the interface, and therefore in the volume of material between the components of the system.

Results of the research. When studying the material of unfilled resin ED-20 at frequency of $\nu=1$ kHz three areas of relaxation were experimentally determined (Table 1). It

was assumed that these relaxation areas are caused by the mobility of groups of macromolecules (γ -transition) in the range $\Delta T=323\dots 373$ K, segments that are parts of the chain in the range $\Delta T=343\dots 393$ K and chains as a whole in the range $\Delta T=373\dots 433$ K. such relaxation processes on the temperature dependence of dielectric losses of the studies specimens prove that during formation of epoxide matrixes the mobility of various types of kinetic units in adsorption and subsurface layers of the polymer have been detected. It is known [6, 7] that during the material formation there appears a surface layer with the properties different to the ones of polymer in volume. As a result of its non-homogeneous, the areas with different density are observed both near the filler surface and at certain distance from it. The study results show (Table 1) that using of original and ESHT modified of epoxide resin ED-20 as a matrix causes manifestation of three relaxation processes characteristic for molecular mobility in the adsorption and subsurface layers. The obtained results make it possible to state that in such polymers, two types of segment activity exist in the surface layer: segments and groups situated in the adsorption layer and segments of groups in the surface layers remoted from filler. Besides, the research results point out that alongside with groups and segments, single chains may take part in relaxation processes.

Table 1

Temperature characteristics of relaxation processes in original and modified epoxy matrix

Com- ponents of matrix	ESHT processing	Process of groups relaxation		Process of segments relaxation		Process of chain relaxation	
		Width of the process, ΔT , K	Temperature of maxima, T_m , K	Width of the process, ΔT , K	Temperature of maxima, T_m , K	Width of the process, ΔT , K	Temperature of maxima, T_m , K
ED 20	-	10	323	19	353	48	383
ED 20	+	19	363	12	383	15	413
ED 20 DEG – 1	-	-	-	20	373	9	403
ED 20 DEG – 1	+	16	343	-	-	30	433

Note.

Concentration of matrix ingredients: ED-20 is 100 m/v, DEG-1 is 10 m/v, PEPA is 11 m/v; + is ESHT processing of material; – is unprocessed material

It is established that after treatment of the ED-20 oligomer with an electric spark hydraulic impact, the temperature of the dielectric loss maxima (ϵ'') shifts to the region of higher temperatures for all determined relaxation processes by 30... 40 K. In addition, the width of the maximum, which is characteristic of the process of relaxation of groups increases after ESHT by 9 K, and for the process of relaxation of segments and main chains of macromolecules, its width decreases significantly [8]. These results of the analysis of studies before and after treatment of the oligomer of the ESHT epoxy resin allow to propose new modes of material formation after such treatment. The results of the research confirm the assumption that after ESHT treatment of resin most of the oligomer transfer into the state of surface layers, and the subsurface layers become not only more saturated segments of macromolecules, but also their total volume increases. By this, a positive contribution of the pre-treatment of the components of ESHT matrix on the properties and the degree of crosslinking of the CM matrix have been established.

The above research results indicate the relaxation of segments and groups of macrochains in areas of the surface layer. In this regard, it was interesting to study the dependence of the loss factor (ϵ'') on temperature for epoxy matrices containing DEG-1

plasticizer. It is found that the introduction of a plasticizer into an epoxy oligomer leads to a slightly different mechanism of structure formation of the composite. It is revealed that introduction of a plasticizer into an epoxy oligomer causes slightly different mechanism of structure formation of the composite. It is shown (Table 1) that compared to raw oligomer ED-20 (not ESHT processed), on temperature dependencies of dielectric losses were not observed on areas responsible for relaxation of groups (γ -transition). In addition, the width of the maximum of the loss factor (ϵ'') of the relaxation process of the segments almost does not change (increases by 1 K), but the displacement of its maxima in the area of higher temperatures occurs by 20 K compared to the original untreated and unplasticized oligomer. This indicates that when crosslinking in the process of structure formation involves a larger amount of oligomer ED-20, as well as molecules of aliphatic resin DEG-1. Besides, it should be noted that in this comparative context, the behavior during temperature relaxation of a plasticized epoxy matrix with a simultaneously modified ESHT epoxy oligomer is of an interest. When analyzing the dependence of dielectric losses of the specified epoxy composite on the temperature, the absence of the area responsible for relaxation of segments was experimentally established (Table 1). Note that the maximum temperature characteristic of relaxation process of the groups is shifted to the area of lower temperatures (20 K), and the maximum temperature responsible for the relaxation process of the main chain is shifted to the area of higher temperatures (20 K) compared to these characteristics based on epoxy resin modified by ESHT. Data analysis suggests that in such a studied material there is a sufficiently high degree of gelation, while the rate of structure formation is quite high in the initial hardening stages.

The next stage was the study of CM filled with dispersed particles of different chemical and magnetic nature and modification of ESHT about the properties of epoxy composites. In Fig. 1... 3, the results of studies of dielectric losses from the temperature of CM containing different concentrations of fillers (alumina, chromium oxide, carbon black) after modification of ESHT epoxy resin, which was subsequently plasticized with aliphatic resin DEG-1 (10 m/v per 100 of ED-20 m/v) are presented. For comparison, these graphs also show the results of curves of dependence of the loss factor (ϵ'') on the temperature of CM, containing a disperse filler based on a plasticized but unmodified ESHT matrix. Analysis of relaxation areas of groups, segments and chains of the matrix is given in table. 1. It has been experimentally established that in general three sections can be distinguished on the curves of the dependence of dielectric losses (ϵ'') on temperature. It is shown (Fig. 1, Table 1) that the introduction of alumina (50 parts by weight per 100 parts by weight of binder) in the unmodified ESHT matrix leads to the appearance of three maxima of the relaxation process of groups, segments and chains. In this case, pre-treatment of ESHT epoxy oligomer in a plasticized matrix with the subsequent introduction of the filler at different concentrations shows a significant shift of the maxima of relaxation processes in the region of higher temperatures [9, 10]. This indicates an improvement in the adhesion interaction at the interface «oligomer-disperse filler». In our opinion, this is due to the activation of macrochains and free radicals during ESHT processing. Such free radicals have greater activity and mobility compared to the original macrochains, which provides a significant interaction with the active groups on the surface of alumina particles. Note that when filling the matrix with alumina at concentration of 30 weight part per 100 parts by weight part of binder, causes a significant increase in the maximum loss factor (ϵ'') to 0.32 (in the area of relaxation of the segments) and to 0.59 (in the area of relaxation of the chain). Such a sharp increase in the dielectric loss indicates high mobility in the surface layers of macromolecules. This indicates a low degree of crosslinking of the matrix around the filler and in the volume of the polymer, which is obviously due to the insufficient concentration of dispersed particles in the system. Accordingly, to increase the cohesive characteristics of these CM, the concentration of additives should be increased. It is shown (Fig. 1, Table 1) that introduction of 50... 80 weight part of alumina in the matrix leads to

decrease in the maximum dielectric loss, i.e. to decrease in mobility on one side, as well as to a more uniform layer-by-layer distribution of segments in the volume of the surface layers. Note that, in our opinion, the optimal concentration is the particles content is 50 m/v per 100 m/v of ED-20, because then the segments are more evenly distributed in the volume of the surface layers.

A slightly different pattern was observed when the untreated plasticized matrix was filled with chromium oxide paramagnet. It is experimentally established that the introduction into the original composition of 50 m/v chromium oxide leads to the appearance of one maximum of dielectric losses, which corresponds to the relaxation of segments in the adsorption layer (Fig. 2, Table 1). The indicators of the loss factor (ϵ'') of the specified maximum is quite large and is 0.40.

Modification of the ESHT epoxy oligomer followed by introduction of the filler at the same concentration leads to the appearance of three maxima of segments relaxation and groups of macromolecules. However, we note that the mobility of groups and segments in such layers is not high enough and is $\epsilon'' = 0.05$ (when analyzing the relaxation process of the main chain). This indicates a fairly high degree of crosslinking and the percentage of gelation in the surface layers of the CM with the specified filler. Subsequently, the introduction of chromium oxide at concentrations of 30 and 80 m/v per 100 m/v of the binder also causes a decrease in the loss factor and the manifestation of relaxation maxima (Fig. 2).

Analysis of the results of studying the curves of dependence of dielectric losses on temperature, given in Table 1, allows to state that the optimal concentration of the specified filler in the system is 30... 50 m/v per 100 m/v of binding agent. Introduction of chromium oxide at this concentration provides more even distribution of segments throughout the volume of the surface layer formed around the filler. Increasing concentration of filler in the system leads to decrease in the width of the maxima, which, in turn, increases the mobility of macromolecules due to increasing temperature, which is the result of low cohesive characteristics of the studied composites.

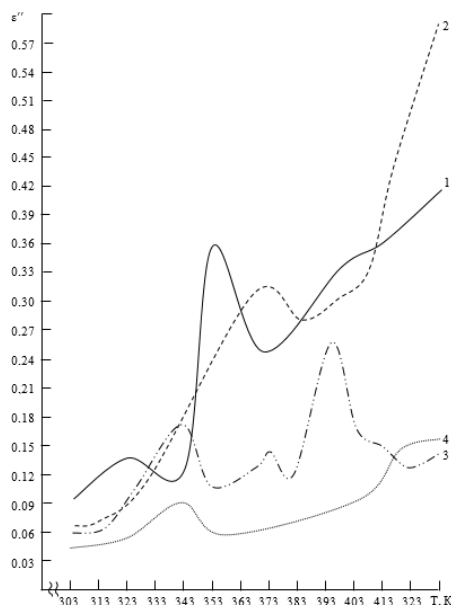


Figure 1. Temperature dependence of dielectric losses (ϵ'') of epoxy composites containing alumina: 1 – without processing (50 m/v); 2 – after ESHT processing (30 m/v); 3 – after ESHT processing (50 m/v); 4 – after ESHT processing (80 m/v)

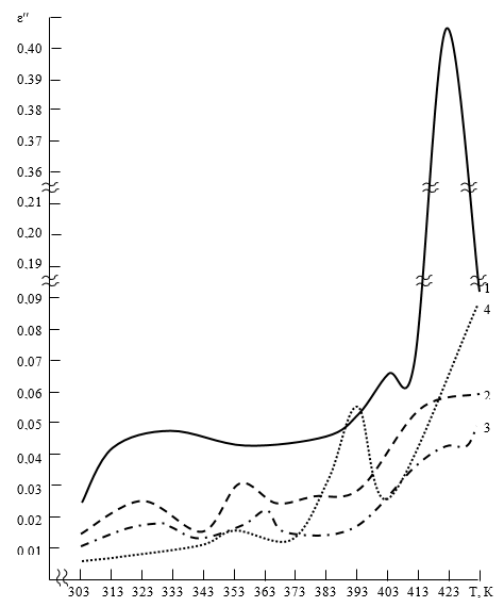


Figure 2. Temperature dependence of dielectric losses (ϵ'') of epoxy composites containing chromium oxide: 1 – without processing (50 m/v); 2 – after ESHT processing (30 m/v); 3 – after ESHT processing (50 m/v); 4 – after ESHT processing (80 m/v)

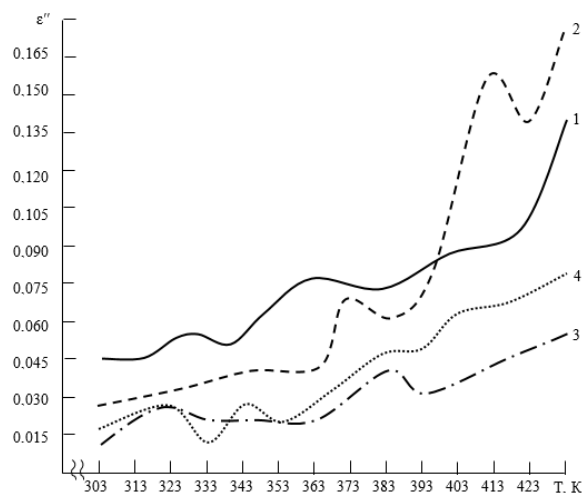


Figure 3. Temperature dependence of dielectric losses (ϵ'') of epoxy composites containing gaseous carbon black: 1 – without processing (50 m/v); 2 – after ESHT processing (30 m/v); 3 – after ESHT processing (50 m/v); 4 – after ESHT processing (80 m/v)

From a scientific and practical point of view, it is interesting to study the processes of relaxation of groups and segments in the surface layers of composites containing ferromagnet gas black (GB). It is found (Fig. 3, Table 2) that the temperature shift of the maximum characteristic of the groups relaxation in the second surface layer, depending on the concentration of the filler is practically not observed and is 323 K. After ESHT modification of epoxide, a shift in the temperature of the maximum was observed, which is responsible for relaxation of segments which are the parts of macromolecules in both directions along the temperature axis, depending on the concentration of dispersed particles. It is shown (Table 2) that at a concentration of additives of 80 m/v there is a shift in the temperature of the maximum of the specified relaxation process in the area of lower temperatures (20 K) after ESHT modification of the epoxy oligomer. Introduction of GB with a content of ED-20 30... 50 m/v per 100 m/v causes a similar shift of the maximum ϵ'' in the area of higher temperatures (10 and 17 K, respectively). This is due to the overlap of segments and groups relaxation areas in the surface layers around the additives at the indicated concentrations in CM. Therefore, such composites are characterized by a more uniform distribution of segments in the adsorption and near-surface layers around the filler in the composition. Introduction of fillers at higher concentrations leads to formation of material with rather rigid structure in the surface layers, especially the adsorption one [11].

Thus, the analysis of the research results shows the effect of particles on the physicochemical interaction of the surface, which allows to experimentally determine the optimal concentration of additives in the volume of composites. Note that when evaluating these characteristics, both the width of the peak dielectric loss and its height on the curve 'loss factor – temperature' should be taken into account. Only with such comprehensive approach it is possible to assess the effect of surface activity and filler concentration on the degree of crosslinking of the matrix in the surface layers and the length of the latter.

It is known [1] that physical modification, as well as chemical grafting of oligomers to the filler surface provides improved physical interaction at the 'polymer-filler' phase boundary. This, in turn, causes a decrease in internal stresses in the surface layers around the filler. In this regard, it was interesting from a scientific and practical point of view to conduct the studies of molecular mobility in the surface layers around the filler, which was introduced into the matrix after pre-heat treatment according to the modes described in the method.

Table 2

Temperature characteristics of relaxation processes in original and modified epoxy composites

Filler	m/v	Process of groups relaxation		Process of segments relaxation		Process of chain relaxation	
		Width of the process, ΔT , K	Temperature of maxima, T_m , K	Width of the process, ΔT , K	Temperature of maxima, T_m , K	Width of the process, ΔT , K	Temperature of maxima, T_m , K
Al ₂ O ₃ (without ESHT treatment of resin)	50	15	323	13	353	24	433
Al ₂ O ₃	30	-	-	17	373	16	433
Al ₂ O ₃	50	16	343	11	373	12	393
Al ₂ O ₃	80	18	343	-	-	30	433
Cr ₂ O ₃ (without ESHT treatment of resin)	50	-	-	-	-	10	423
Cr ₂ O ₃	30	17	323	10	353	56	433
Cr ₂ O ₃	50	16	330	22	363	18	433
Cr ₂ O ₃	80	-	-	18	353	28	393
GB (without ESHT treatment of resin)	50	21	323	11	363	16	433
GB	30	-	-	15	373	11	413
GB	50	15	323	10	380	10	433
GB	80	16	323	8	343	22	433

This physical modification of the dispersed particles will allow to activate the hydroxyl and other groups on the surface of the additives blocked by hydrogen bonds before the physicochemical interaction. This, in turn, will improve the interfacial interaction, which in our opinion, will be manifested in the length and relative content of segments in the subsurface and adsorption layers.

It has been experimentally established that physical temperature modification of the dispersed filler affects the molecular mobility in the surface layers in different ways (Table 3). In particular, it is shown that GB heat treatment does not provide a shift in the maxima of the dielectric losses during the process of relaxation of groups. However, the maximum ϵ'' in the process of segment relaxation is shifted to the region of higher temperatures by 20 K. It is proved that such a sharp change occurs due to the activation of physical and mechanical processes after heat treatment of the filler, which is associated with the active influence of phase separation of 'filler-polymer' boundaries.

A slightly different picture was observed after heat treatment of chromium oxide and aluminum. In particular, when modifying chromium oxide, a decrease in the maximum loss factor, which is responsible for the relaxation process of the groups, was not detected. Obviously, when the concentration of the filler (50 m/v per 100 m/v of binder) is introduced, after the heat treatment of the filler, a strongly crosslinked polymer structure is formed in the surface layer, which is specific by its considerable length. In this case, the entire volume of the polymer turns into the state of the surface layer.

In the case of heat treatment of alumina, it is found that the temperature shift of the maxima after thermal modification of the filler occurs in the direction of lower temperature on the time axis (in the analysis of the relaxation processes of the segments). This, in turn, indicates the formation of polymer structure with a high degree of crosslinking in the surface layer, but not as rigid as in the case of filling CM with chromium oxide. Accordingly, such composites are characterized by less stressful state and better relaxation properties.

Table 3

Temperature characteristics of relaxation processes in modified composites during pre-treatment ($T = 423$ K) of filler

Filler	Heat treatment of fillers at 423 K	Process of groups relaxation		Process of segments relaxation		Process of chain relaxation	
		Width of the process, ΔT , K	Temperature of maxima, T_m , K	Width of the process, ΔT , K	Temperature of maxima, T_m , K	Width of the process, ΔT , K	Temperature of maxima, T_m , K
Al ₂ O ₃	+	21	348	12	363	11	403
Al ₂ O ₃	-	16	343	11	373	12	393
Cr ₂ O ₃	+	-	-	23	383	23	433
Cr ₂ O ₃	-	16	330	22	363	18	433
GB	+	20	323	14	393	10	433
GB	-	15	323	10	380	10	433

Note:

Concentration of the filler is 50 m/v per 100 m/v of epoxy resin.

+ is heat treatment of the filler; - is unprocessed filler.

Conclusions. The results of the research show that the preliminary modification of the fillers allows to regulate the properties of the material in the surface layers depending on the nature of the filler. In most cases, this modification provides a more equilibrium state of the polymer in the surface layers, which allows to develop a method of predictive control of the performance of heterogeneous systems.

Herewith, the results of the research show the importance of conducting an electrospark hydraulic impact of an epoxy oligomer followed by hydrodynamic combination of components with a plasticizer (aliphatic resin DEG-1 and filler). This step-by-step creation of a matrix with the subsequent introduction of dispersed particles and a hardener provides the formation of composites with high cohesive strength due to the creation of surface layers of considerable length and with a high degree of crosslinking. Dielectric loss studies have shown that three areas of the groups relaxation, segments and chains of matrix macromolecules were observed in composite materials at different stages of temperature increase, respectively. The obtained relaxation areas were interpreted by analyzing the relaxation processes of different kinetic units of the epoxy binder, which correspond to the regions of their location on the curves of temperature-time dependences of the dielectric loss angle tangent. This, in turn, allows to predictably regulate the processes of structure formation in the material by selecting the optimal concentration of the filler and temperature-time modes of processing of the epoxy composite.

In our opinion, the approach to the regulation of the structure in the surface layers around the filler by its preliminary heat treatment in a thermal field at certain modes is promising. This physical modification, depending on the nature of the filler, can affect the length of the surface

layers. This, in turn, allows to predictably create a more rigid structure of the polymer, as well as a material of considerable length with relatively homogeneous surface layers.

In further researches, the structural transformations in the material containing two types of binders that are able to form polymer-polymeric systems are to be investigated.

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МОЛЕКУЛЯРНА РУХЛИВІСТЬ ЕПОКСИДНОГО В'ЯЖУЧОГО У МОДИФІКОВАНИХ ЕЛЕКТРОІСКРОВИМ ГІДРОУДАРНОМ КОМПОЗИТАХ

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Резюме. У роботі при дослідженні молекулярної рухливості у поверхневих шарах методом визначення діелектричних характеристик матеріалів, модифікованих електроіскровим гідравлічним ударом, визначено оптимальний вміст дисперсного наповнювача. Представлено порівняльні дані тангенса кута діелектричних втрат оброблених та необроблених композиційних матеріалів. Результати досліджень показують, що попередня модифікація наповнювачів дозволяє регулювати властивості матеріалу в поверхневих шарах залежно від природи наповнювача. У більшості випадків така модифікація забезпечує рівноважніший стан полімеру в поверхневих шарах, що дозволяє розробити метод прогнозного контролю продуктивності гетерогенних систем. Фізична модифікація дисперсних частинок дозволить активувати гідроксильні та інші групи на поверхні добавок, блокованих водневими зв'язками перед фізико-хімічною взаємодією. Це, у свою чергу, покращить міжфазну взаємодію, що, на нашу думку, буде проявлятися у довжині та відносному вмісті сегментів у приповерхневому та адсорбційному шарах. Доведено, що різка зміна відбувається за рахунок активації фізико-механічних процесів після термічної обробки наповнювача, що пов'язано з активним впливом фазового поділу кордонів «наповнювач-полімер». Дослідження діелектричних втрат показали, що на різних стадіях підвищення температури в композитних матеріалах спостерігалися три області групової релаксації, сегменти та ланцюжки макромолекул матриці. Отримані зони релаксації інтерпретували шляхом аналізу релаксаційних процесів різних кінетичних одиниць епоксидного в'язучого, які відповідають ділянкам їх розташування на кривих температурно-часових залежностях тангенса кута діелектричних втрат. Це дозволяє прогнозувати регулювати процеси структуроутворення в матеріалі шляхом підбору оптимальної концентрації наповнювача та температурно-часових режимів обробки епоксидного композиту.

Ключові слова: електроіскровий гідроудар, молекулярна рухливість, композит.

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