Досліджено вплив високодисперсних наповнювачів на механічні характеристики епоксикомпозитних матеріалів, які використовуються в якості різнофункціональних шарів захисних покриттів. Досліджено особливості процесу структурування епоксикомпозитів з високодисперсними частинками, композиції яких було піддано впливу ультразвукової обробки та електромагнітного поля. Доведено позитивний вплив обробки композицій у фізичних полях, що дозволило значно підвищити адгезійну міцність епоксикомпозитів, що містять високодисперсні частинки

Ключові слова: епоксидна композиція, ультразвукова обробка, електромагнітне поле, високодисперсний наповнювач, залишкові напруження

Исследовано влияние высокодисперсных наполнителей на механические характеристики эпоксикомпозитных материалов, используемых в качестве разнофункциональных слоев защитных покрытий. Исследованы особенности процесса структурирования эпоксикомпозитов с высокодисперсными частицами, композиции которых подверглись воздействию ультразвуковой обработки и электромагнитного поля. Доказано положительное влияние обработки композиций в физических полях, что позволило значительно повысить адгезионную прочность эпоксикомпозитов, содержащих высокодисперсные частицы

Ключевые слова: эпоксидная композиция, ультразвуковая обработка, электромагнитное поле, высокодисперсный наполнитель, остаточные напряжения

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EXAMINING THE EFFECT OF PHYSICAL FIELDS ON THE ADHESIVE STRENGTH OF PROTECTIVE EPOXY COMPOSITE COATINGS

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1. Introduction

Epoxy composite material sare widely used as protective coatings on the surfaces of metallurgical, machine-building and instrument-making equipment [1]. These numerous details operate under the influence of variable temperatures, stresses and strains. The main shortcoming of the given coatings is the complexity of the formation of a homogeneous structure, which leads to the deterioration of the physical and mechanical properties of coatings, including their adhesive strength [2]. Adhesive interaction between a protective coating and the surface of a substrate is one of the main factors that determine durability and working ability of parts of machinery [3].

Application of active finely dispersed additives makes it possible to modify the spatial grid of a polymer binder at the micro structural level in order to improve the mechanical characteristics. The prospects of obtaining, examining the properties and utilization of finely dispersed and nanoscale powders are widely discussed in the world science as their application will enable significant progress in materials science and the physics of solid body [4]. Reducing dimensions of the powder particles to nano-meters makes it possible to improve mechanical characteristics of structural materials. Their use also contributes to a change in the physical properties of composites [5]:

– lattice parameters;
– electron structure;
– temperature of melting and phase transitions;
– the Debye and Curie temperature;
– the speed of diffusion and chemical reactions.

Small dimensions, chemical and diffusion activity, high melting temperatures of the particles allow the use of highly dispersed powders of metals or compounds as a promising filler for obtaining polymer-composite coatings [6]. Given this, it is a relevant task to develop epoxy composite protective coatings that possess high adhesive strength to the surface of highly dispersed particles.

2. Literature review and problem statement

Polymer composite materials with highly dispersed fillers based on epoxy binders are characterized by high adaptability when applied on the parts with a complex surface profile. These materials are also distinguished by high operational characteristics under the influence of aggressive environments [7], however, due to low impact toughness, their use is limited. The effectiveness of modification of the thermoreactive polymers with highly dispersed particles depends on the following characteristics of filler: the size and shape of the particles, their volumetric content, particle distribution by size, uniformity in the distribution of filler in a polymer matrix [8], which is difficult to achieve when structuring the epoxy composites under normal conditions. Due to the high surface energy, highly dispersed particles have a tendency to the formation of agglomerates and sedimentation, which complicates their uniform distribution in a polymer and reduces mechanical characteristics of polymer-composites. It is related to the processes of selective adsorption interaction between a polymer matrix and a filler that leads to instability, changeability in a free state, rapid oxidation and uneven distribution of finely dispersed particles in a polymer matrix [9–10].

Among the methods of adjusting the structure and improving the properties of composites, of relevance is the formation of polymer-composite materials under the influence of external energy fields [11]; however, the patterns of influence of external factors on the mechanism of structure-formation in composites with highly dispersed and nano-fillers have not been sufficiently examined. The application of the given fields leads to a rise in the temperature of a binder, which significantly reduces its viscosity. Accordingly, a polymeric composition better fills irregularities and defects at the surface of particles, which is a necessary condition for the formation of strong bond between a polymer and a solid phase [12]. The article has not considered magnetic, chemical, kinetic and thermodynamic activity of the filler at external impact, which requires studying the chemical and physical-mechanical phenomena that occur during formation of the material. In addition, the rise in temperature leads to the emergence in a reactive polymer of free functional groups (hydroxyl, carboxyl, carbonyl), which increases the number of physical and chemical bonds between them and the surface of the solid phase. Treating the compositions with physical fields at the stage of mixing contributes to the uniform distribution of components in the volume of composite due to the enhancement of their mobility [13]. Physical fields act at the level of a substructure, they change a conformational set of molecules, which enables the creation of optimal spatial grid of the binder. As a result of this, there occurs the improvement of physical-mechanical and operational characteristics of composite materials and protective coatings based on them [14]. That is why there is an unresolved issue of examining the influence of physical fields on the distribution of highly dispersed fillers of various physical (dia-, para- and ferromagnetic) and chemical nature in an epoxy matrix. In this case, there is a need for a detailed study of the processes of distribution of components of an epoxy composite system and their interaction under the influence of physical fields at the stage of structuring the composition.

3. The aim and objectives of the study

The goal of present research is to study the processes of structuring the epoxy composite materials filled with highly dispersed particles through controlled impact on the compositions of physical fields. This will improve adhesive strength, physical-mechanical and operational characteristics of the modified epoxy composite coatings of optimal composition.

To accomplish the set goal, the following tasks are to be solved:

– to determine adhesive strength of the epoxy composite materials filled with highly dispersed powders without treatment of the composition;

– to examine the influence of physical fields on adhesive strength and residual stresses of the epoxy composite materials filled with highly dispersed powders;

– to analyze the microstructure of epoxy composite materials filled with highly dispersed powders with treatment of the composition in physical fields.

4. Procedure of treating the composition and examining the adhesive strength of epoxy composite coatings

We used the epoxy resin of grade ED-20 (GOST 10587-84) as a matrix of polymer-composites, which is a liquid reactive oligomeric product based on diglycidyl ether of diphenyl propane. Epoxy-dianova resin ED-20 is a viscous liquid without visible mechanical inclusions, which dissolves in toluene. When the resin is heated to 873 K and higher, a small amount of volatile substances is released (epichlorohydrin and toluene). Coatings based on the resin ED-20 possess high adhesion to different materials, high hardness, elasticity, high dielectric properties, resistance to aggressive environments.

To harden the epoxy compositions, we used polyethylene polyamine (PEPA) (TU 6-02-594-70), which is a liquid with low viscosity. The process of structuring envolves a chemical interaction between the final amine groups of a hardener and the macro molecules of epoxy resin at room temperature and lowered temperatures under conditions of elevated humidity.

We exploited powders of different physical and chemical nature with a dispersion of 0.25-500 μm as additives.

Iron powder of the type PZhR-3 (GOST 9849-86) contains in its composition not less than 97 % of primary substance, particle dispersion is 30–50 μm.

Carbonyl iron of the type R-20 (GOST 13610-79) is a finely dispersed powder of pure iron with an average diameter of particles of 2.5–5 μm. The powder has elevated electrical conductivity, resistance to aggressive environments, and is also characterized by high plasticity indicastors. It contains particles of spherical shape with dense structure free from sharp protrusions.

Zirconium powder of the type PTsrK1 (TU 48-4-234-84) has an average size of particles of 1–2 μm. Zirconium has a high corrosion resistance to atmospheric gases, water and alkilis, it does not react with hydrochloric and sulfuric (concentration of up to 50 %) acids, it is plastic and demonstrates paramagnetic properties.

Fullerene black (TU 2166-004-65523364-2010) is the fullerene soot, received after the removal of mixtures of fullerenes by non-polar organic solvents. The powder is not soluble, total content of fullerenes does not exceed 0.10 %.

Ftoroplast powder of the type 4PN-20 (GOST 10007-80) is resistant to all acids, solvents, petroleum products, alkalis in the working range of prolonged operation from –542 K to +533 K. This powder is resistant to water vapor and climatic impacts. The powder particles have high dielectric and antifriction properties. The average particle size is 6–20 μm.

Study of adhesive strength of epoxy composite coatings was conducted by procedure in line with GOST 14760-69. The procedure is based on measuring the tear resistance of adhesive joints of the face surfaces of steel rods with conical projections for the self-centering in grippers of the tensile testing machine Zwick RSA100 (manufactured in Germany). The rate of change in loading was 2 mm/min. The front surface area in the place of glueing the rods was 2 cm^2.

The formation of experimental samples implied obtaining a homogeneous composition, which contained the required components. Depending on the volume of the samples we calculated a quantitative content of components in mass parts per 100 mass fractions of the epoxy resin ED-20. The composition was formed by introducing the hardener PEPA (12 mass fractions per 100 mass fractions of ED-20) and the fillers to the epoxy resin. The composition was mechanically agitated at each stage to provide high homogeneity of the system. At the next stage, the composition was exposed to the influence of physical fields. The formed composition was applied onto a prepared surface or poured into special moulds. The surface was pre-treated with an abrasive to achieve the required roughness and was degreased with a solvent.

Since mechanical properties of the polymer composites depend on the technology of formation, then so much attention is paid to studying the influence of external physical fields on the processes of structuring. For the uniform distribution of a highly dispersed filler in the volume of a polymer matrix, the compositions, after mechanical agitation, were treated with ultrasound or in electromagnetic field. The ultrasound treatment was carried out in a container of laboratory installation (Fig. 1) in the water medium at frequency 20 kHz. The prepared composition in a container the size 6×4 cm was placed at a distance of 10 mm from the source of ultrasonic waves. Treatment duration made up 5–10 min.

To study the influence of electromagnetic treatment, we used a laboratory installation that is capable of generating a high-frequency electromagnetic field (Fig. 2). The treatmemt was conducted in the air environment. Treatment duration made up 5–10 min.

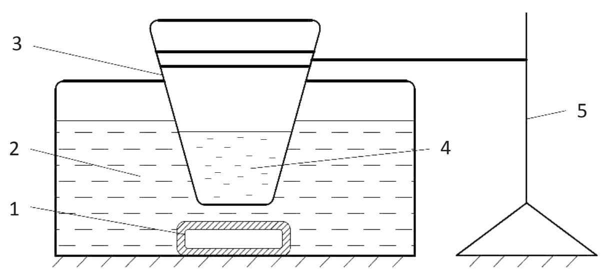


Fig. 1. Schematic of the installation for treating the composition with ultrasound: 1 – the source of radiation; 2 – medium (water); 3 – container; 4 – composition; 5 – tripod

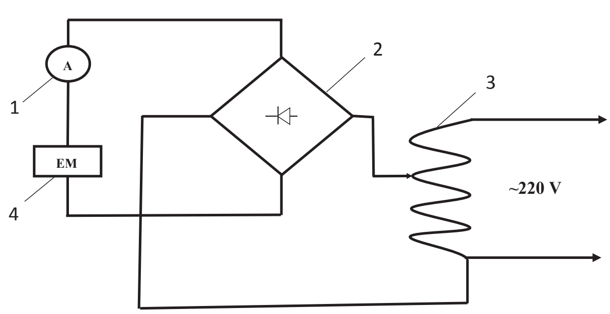


Fig. 2. Schematic of the installation for treating the composition in electromagnetic field: 1 – amperemeter; 2 – diode bridge; 3 – compensator; 4 –electromagnet

Hardening of the epoxy composites under normal conditions lasted for 24 hours. Additional thermal treatment was carried out in a furnace under a stepwise mode: 343 K with curing for 1 h, 373 K with with curing for 1 h, 403 K with curing for 4 hours.

The study of the microstructure of epoxy composites was performed at the raster electronic microscope (EVO 50) (manufactured by Karl Zeiss, Germany).

Internal strains were determined using the console method in line with GOST 13036-67 on the laboratory installation. The method is based on measuring the deviation from the initial position of the free end of the cantilever pinned elastic metal plate. Deformation of the plate takes place under the influence of internal stresses that occur in coatings.

5. Examining the adhesive strength and microstructure of epoxy composite materials

It was experimentally determined (Fig. 3) that the powders of iron PZhR-3 and carbonyl iron P-20 (6–10 mass fractions) reduce the adhesive strength of epoxy composites by 1.6–1.7 times. This is due to the formation of insufficient number of chemical bonds between the macro molecules of an epoxy polymer matrix and the highly dispersed particles. Accordingly, in this case, there forms a structure, which is characterized by a low content of gel fraction (G=91 %). This structure has a large number of defects (Fig. 4, *a, b*):

– air cavities (d=50–150 μm);
– agglomerates (d=10–40 μm).

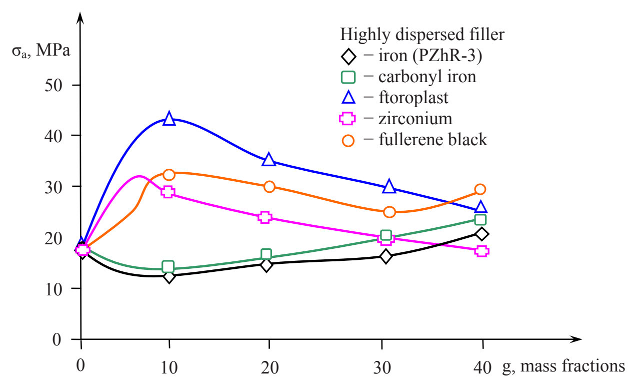


Fig. 3. Dependence of adhesive strength of epoxy composites on the content and type of a highly dispersed filler

When using the powders of types PZhR-3 and R-20, there occurs an increase in the adhesive strength of epoxy composites by 1.4–2.4 times. This increase takes place in the case of application of the higher degrees of filling the system (10–40 mass fractions) compared with a low-filled system (6–10 mass fractions). An increase in the adhesive strength is associated with a decrease in the residual stresses in the material (σ_{res}=2.5 MPa) by 24 %.

The introduction of ftoroplast powder in the amount of 6 mass fractions into an epoxy polymer matrix leads to an increase in the adhesive strength by 2.5 times compared with an unfilled system (σ_a=17.3 MPa). Ftoroplast powder contributes to the formation of a structure of epoxy composites with low residual strains (σ_{res}=1.96 MPa), due to the ability of the system to relax during the process of structuring. This is possible due to the high plasticity of the ftoroplast particles and the formation of a structure with a low content of air inclusions (d=50–100 μm) (Fig. 4, *c*). It was found that the range of optimal content of highly dispersed particles of ftoroplast in a matrix is 6–10 mass fractions, at which the adhesive strength of epoxy composites reaches maximum values. Increasing the content of powder in the composite to 10–30 mass fractions leads to a smooth reduction in the adhesive strength, by 3–15 %. At the content of 30–40 mass fractions, there occurs a sharp reduction in the adhesive strength, by 20–30 %. This is linked to the presence of agglomerates (d=100–200 μm) and other structural defects (pores of d=200–300 μm) (Fig. 4, *d*). These defects were formed due to the uneven distribution of particles of the filler in the volume of the matrix, the result of which is the incomplete wetting of highly dispersed particles with the oligomer.

It was established that the introduction of zirconium powder to an epoxy matrix predetermines a growth in the adhesive strength by 75 % (σ_a=30.3 MPa) at the filler content of 6 mass fractions compared to the unfilled system. Zirconium powder forms strong bonds between a surface of the filler particles and a polymer matrix with minimal residual stresses (σ_{res}=1.48 MPa) in the epoxy composites without treatment. This is possible due to the high capacity of the surface of zirconium particles to wetting by the oligomer and the formation of homogeneous compositions. Increasing the content of zirconium powder to 40 mass fractions reduces the adhesive strength by 50 % (σ_a=15.7 MPa). This is mainly due to the emergence of various defects in the form of air cavities (d=150–200 μm) (Fig. 4, *e*).

Low values of adhesive strength under the condition of increased content of a filler can be also explained by the increased viscosity of the composition, as a result of which the complexes of macromolecules do not fully fill the substrate micro irregularities.

It was establsihed that the introduction of highly dispersed powder of fullerene black to an epoxy composite in the amount of 4–8 mass fractions leads to an increase in the adhesive strength by 1.5 times. This is due to the formation of a system with low residual strains (σ_{res}=1.5 MPa).

The presence of highly dispersed particles in the system leads to a growth in the cohesive strength of composite materials due to the particles' ability to interact intensively with the macro molecules of oligomer. This leads to an increase in the gel fraction (G=95 %) due to the increasing number of chemical bonds in the volume of the polymer.

It was found that the epoxy composition (Fig. 5) is sensitive to the effect of electromagnetic field. The result is a growth in the adhesive strength of epoxy polymer by 13 % compared with the epoxy polymer with composition not treated in the physical field. Maximal adhesive strength (σ_a=70.6 MPa) is demonstrated by the epoxy composite materials filled with the powder of fullerene black under condition of the composition treatment in electromagnetic field in comparison with similarly treated non-filled epoxy polymers. The performed treatment predetermines a uniform arrangement of macromolecules and supramolecular oligomer structures at the surface of dispersed particles. This provides for an increase in the degree of structuring (G=97 %) in the matrix and a decrease in the residual stresses (σ_{res}=0.8 MPa).

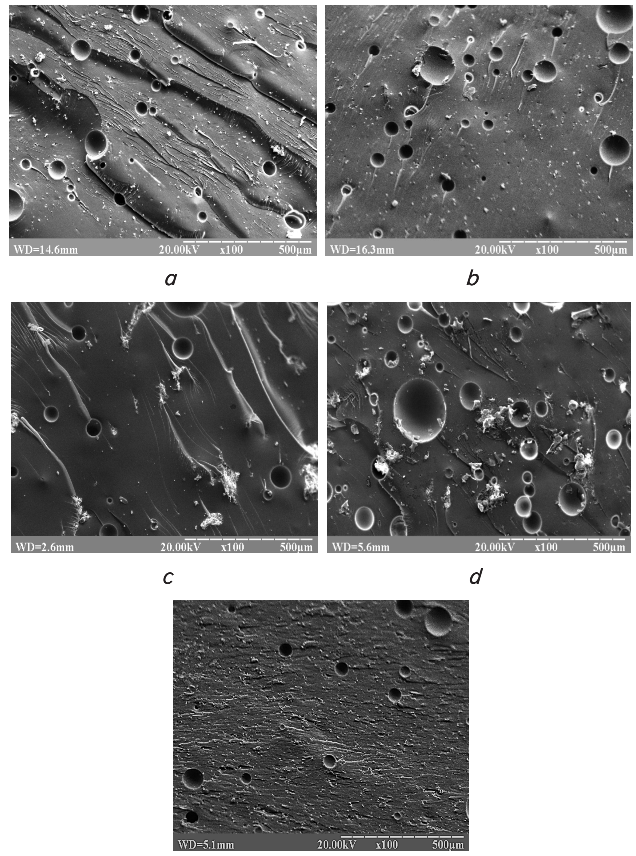


Fig. 4. Fractograms of the fracture of epoxy composites filled with highly dispersed particles: *a* – iron of the type PZh-3; *b* – carbonyl iron; *c* – ftoroplast (6 mass fractions); *d* – ftoroplast (30 mass fractions); *e* – zirconium, ×100

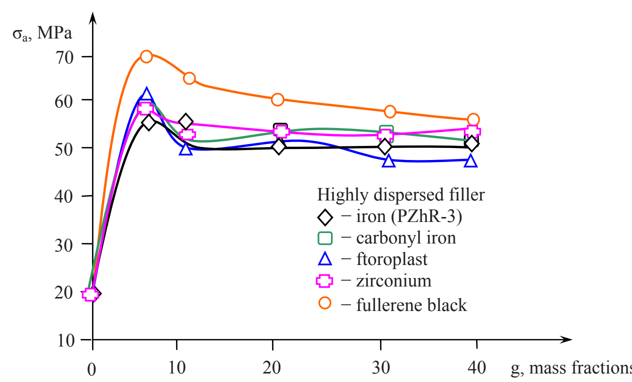


Fig. 5. Dependence of adhesive strength of the epoxy composites whose compositions are modified in the electromagnetic field on the content and type of a highly dispersed filler

It was established that treating the compositions in the electromagnetic field leads to a sharp rise, by 3.1–4.2 times, in the adhesive strength of epoxy composites compared to the non-filled epoxy polymers. The content of highly dispersed powders in the given composites is 6–10 mass fractions. For the epoxy composites with the introduction of powders of iron of the type PZhR-3 and R-20, there occurs a growth of the adhesive strength by 5.2–5.6 times compared to the epoxy composites without treatment. For the composites with zirconium and ftoroplast powders, there occurs a growth of the adhesive strength by 1.5–1.8 times compared to the composites without treatment. Tensile strength of epoxy composites with ftoroplast powder is 63.5 MPa, of epoxy composites with zirconium powder is 56.25 MPa. Treatment of the compositions with highly dispersed particles in the electromagnetic field increases the adhesive capacity because at the surface of the particles the reactive centers are activated due to the action of an external field. The given centers in the process of formation of epoxy composites enable additional formation of the bonds, which provides for an overall improvement of the mechanical properties of epoxy composites.

An analysis of the surface of the adhesive bond revealed that the epoxy composite materials without the pre-treatment (Fig. 6, *a*) possess an adhesive character of destruction, due to a low adhesive capacity of the epoxy composite. The epoxy composites whose composition was treated in the electromagnetic field possess a cohesive character of destruction of the adhesive bond (Fig. 6, *b*), which indicates an increase in the adhesive strength of the system. The influence of electromagnetic waves increases internal energy and entropy of the system. This leads to the activation of the chains of macromolecules with the formation under the influence of irradiation of free radicals with their subsequent recombination in the process of structuring of the epoxy polymer matrix.

a

b

Fig. 6. General view of the surface of adhesive bond of the epoxy composite coatings, filled with highly dispersed zirconium powder, *a* – without treatment, *b* –treatment of the compositions in electromagnetic field

The adhesive strength of epoxy polymers after treating the compositions by ultrasound (Fig. 7) grows by 1.7 times compared with the non-treated compositions (Fig. 3). A growth of the adhesive strength by 1.5 times occurs after treating the compositions in electromagnetic field (Fig. 5). The application of ultrasonic treatment ensures an improvement of the given characteristics, as a result of the intensification of interaction among the components and the reduction in system defectability as a whole. At the surface of epoxy polymers without treatment we registered clouding and uniformity of structure (Fig. 8, *a*). Such defects were not registered in the case of treating a composition with ultrasound (Fig. 8, *b*).

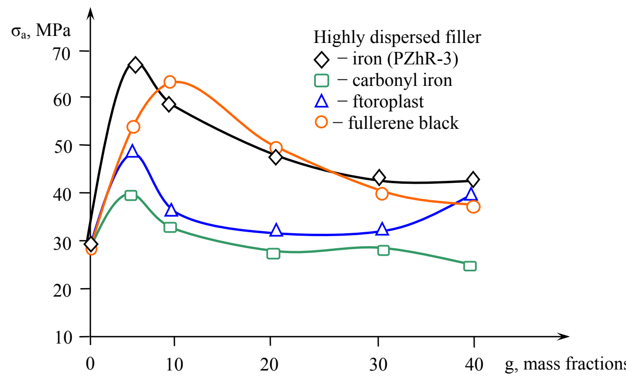
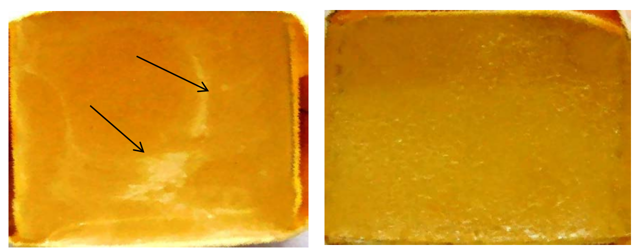


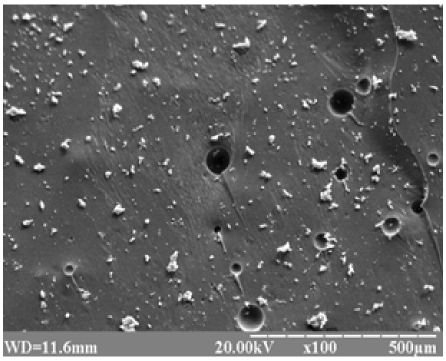
Fig. 7. Dependence of adhesive strength of the epoxy composites whose compositions are modified by ultrasound on the content and type of a highly dispersed filler



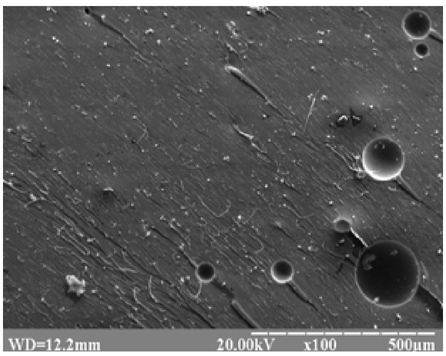
a *b*

Fig. 8. General view of the surface of epoxy polymers: *a* – without pre-treatment; *b* – treating the composition with ultrasound

The treatment of composition with ultrasound contributes to a growth by 1.2 times of the adhesive strength of epoxy composites that contain ftoroplast powder. In the case of using a powder of carbonyl iron, it increases by 3 times. In the case of applying the iron powder of type PZhR-3, the growth is 12.6 times, and using a powder of fullerene black – 3.6 times. Growth occurs for the systems that contain powders of 6–10 mass fractions in comparison to the non-filled epoxy polymers. An increase in the strength of adhesive epoxy composites whose compositions are treated with ultrasound is due to the improved wetting of the particles surface with a viscous oligomer. The increase in the filler content is accompanied by a decline in the adhesive strength, which is caused by the presence of agglomerates (d=30–70 μm) (Fig. 9, *a*), which remained in the system and limit the wetting of the surface of separate particles with an oligomer. This also increases the stressed state of the system (σ_{res}=1.4–2.4 MPa) due to a low capacity of the segments of macromolecules to micro dispalcements in the outer surface layers of the particles. Particles of carbonyl iron are evenly distributed in the volume of the material without the formation of agglomerates (Fig. 9, *b*), indicating the feasibility of treating the compositions with ultrasound.



a



b

Fig. 9. Fractograms of the fracture of epoxy composites whose compositions are modified by ultrasound and filled with highly dispersed particles: *a* — iron of the type PZhR-3; *b* — carbonyl iron ×100

The fracture surface of epoxy composites is characterized by the presence of a small number of air cavities compared with the epoxy composites that were not treated in the physical field. Treating the compositions with ultrasound provides for the formation of a more homogeneous structure of epoxy composites, which is confirmed by obtaining a monoplanar fracture surface without sharp changes in the relief. This indicates the formation of the system with a low stressed state, which is a positive moment for receiving polymer composites with high mechanical and operational characteristics.

6. Discussion of results of examining the adhesive strength of epoxy composites

A special feature when examining adhesion is determining the influence of electromagnetic field and ultrasound on the epoxy composites that contain a hardener in the composition. Previous analogous studies were conducted disregarding the content of hardener in the formulation of a composition. Articles [1–6] reported the introduction of a hardener to the formulation of a viscous composition after treatment in the physical field. Accordingly, this technology does not provide for the penetration of macromolecules of a hardener to the resulting groups of epoxy resin, which are localized in the narrow-gap sites at the surface of particles of the filler. Formation of optimal number of physical-chemical bonds at the surface of particles of the filler is a prerequisite for the provision of high adhesive strength of polymer composites. For a better understanding of the interaction between the components of the system, it is necessary in the future to conduct a study into the microstructure of epoxy composites in the boundary layers at higher magnifications.

Examining the conditions and processes of structuring at the phase interface is an important aspect for understanding the characteristics of the formation of bonds in polymeric systems based on thermosets. This will enable obtaning new polymer composite materials with a set of controlled physical-mechanical and operational characteristics.

In the future, it is planned to conduct experimental research using the nanosized particles, which will make it possible to receive dependences of the influence of the nature, quantity, particle shape on the mechanical and thermal-physical characteristics of polymer composites. It will be possible under condition of the detailed study into processes of structuring in the boundary layers of the system "nanodispersed particle – polymer binder". It is important to conduct experimental research under different modes of treatment in physical fields and under condition of the use of modifiers of a polymeric binder.

7. Conclusions

1. It was established that in the epoxy composites containing iron powders in the amount of 6–10 mass fractions, there occurs a decrease in the adhesive strength by 1.6–1.7 times compared with the non-filled system. This is due to the complexity of the formation of chemical bonds at the highly developed particle surface. With an increase in the content of the given fillers, the adhesive strength increases by 1.4–2.4 times compared with the low-filled system (6–10 mass fractions). The complexity of the formation of bonds is compensated for by an increase in the number of bonds between the total surface of the particles. In the case of using non-metallic highly dispersed particles in small quantities, the adhesive strength grows by 27–58 % due to the better wettability of particles. With a growth in their number, this characteristic is reduced by 34–40 % due to the emergence of agglomerates of the particles.

2. We established that treating the compositions in the electromagnetic field predetermines the growth by 3.1–4.2 times of the adhesive strength of epoxy composites compared to a non-treated system. This applies to epoxy composites that contain highly dispersed powders of zirconium, fullerene black and ftoroplast in the amount of 2–6 mass fractions. This occurs due to the formation of additional physical-chemical bonds between a polymer matrix and the surface of particles with high specific energy. In the case of treatment in the electromagnetic field of the epoxy compositions filled with iron powders (of the type PZhR-3 or R-20) in the amount of 6–10 mass fractions, it leads to an increase by 5.2–5.6 times in the adhesive strength of epoxy composites. This is due to the activation of free radicals of macro molecules, which intensively interact with hydroxyl groups at the surface of metal particles, resulting in a significant growth of the number of physical-chemical bonds (G=99.7 %). External physical fields reduce the viscosity of oligomer and provide for a more uniform distribution of particles, the result of which is a reduction of the strained state of the system by 65 %.

3. In the epoxy composites without treatment filled with the iron powder, there forms the structure characterized by

a large number of defects: air cavities (d=50–150 μm) and agglomerates (d=10–40 μm). This is explained by a high viscosity of the composition, which limits the wettability and reduces the adhesive strength. At small number of ftoroplast particles, there forms the structure with a low content of air cavities (d=50–100 μm). In the case of growth of the content of a filler, the agglomerates emerge (d=100–200 μm), as well as defects of the structure (air cavities d=200–300 μm). Applying the treatment of compositions in physical fields significantly reduces the number of air cavities, by 5–7 times, their size – by 3–4 times, and prevents the formation of agglomerates. This is possible by increasing the mobility of segments of macromolecules and by enhancing their reactivity.

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