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THE INFLUENCE OF ULTRASONIC TREATMENT ON THE MECHANICAL PROPERTIES OF EPOXY COMPOSITES MODIFIED WITH FINE POWDER OF TITANIUM OXIDE

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Abstract. *The use of physical modification of the structure of epoxy composites by ultrasonic treatment of compositions at the stage of mixing of components is substantiated. The results of investigations of the properties of epoxy composites with different degree of filling of the system under the influence of high-frequency ultrasonic vibrations are presented. The influence of the degree of filling of the system and the duration of the ultrasonic treatment of the compositions on the adhesive strength, compressive strength and impact energy of epoxy composites were analyzed. The optimal duration of exposure of compositions under the influence of ultrasonic vibrations is determined. The positive effect of ultrasonic treatment on the structure and properties of epoxy composites filled with fine powder of titanium oxide is proved. The content of titanium oxide powder in the epoxypolymer matrix is optimized, that provides the mechanical characteristics of epoxy composites. Developed epoxy composites should be used as a protective coating of equipment that is exposed to corrosive environments.*
Keywords: *epoxy composite, adhesion strength, compressive strength, impact energy, ultrasonic treatment.*

Introduction. Nowadays improvement of polymeric materials consists not only in the development of the chemical composition of new polymers, but in most cases in the modification of the structure of "classical" polymers. The modification is to change the structure and, accordingly, the properties of the polymer at different levels of the technological process. The purpose of the modification is to improve the technological, mechanical and operational properties of polymers: increasing the viability, reducing the viscosity, increasing the strength, thermal, bio- and chemical resistance, increasing the dielectric properties, reducing the flammability. Depending on the nature of the external influence, the modification methods are divided into chemical, physico-chemical and physical. The most technological are the physical modification methods: heat treatment, modification by ray-methods, periodic deformation, polymer processing in electric and magnetic fields. Physical modification can be carried out at various stages of production, processing and application of polymers: during synthesis, at the stage of processing the polymer into a product, during processing of the finished material or during its operation. Combining physical modification with chemical or physico-chemical methods modification of polymers is quite effective [1-4].

Among the methods of modifying the structure and improving the properties of polymers based on reactoplasts, the formation of polymer composite materials under the influence of external energy fields is relevant. However, the regularities of the influence of external factors on the mechanisms of structure formation in composites with highly dispersed fillers have not been sufficiently studied. The use of external fields lead to an increase the temperature of the binder, which significantly reduces its viscosity. Accordingly, the polymeric binder better fills in the irregularities and defects on the surface of the particles, which is a prerequisite for the formation of stronger bonds between the polymer and the solid phase. The treatment of the compositions by physical fields at the mixing stage promotes an even distribution of the components in the volume of the composition by increasing their mobility. Physical fields operate at the substructure level; they change the conformational set of molecules, which allows forming optimal parameters of the spatial grid of the polymer matrix. As a result, there is an increase in the physical and mechanical and operational characteristics of polymer composite materials and protective coatings [5].

Among the wide range of existing polymers, epoxy resins, which due to their high adhesion and processability, are widely used in the technique for forming the epoxy composite products or coating [4], occupy a significant place. These epoxy resins are convenient for the introduction of additives and physical modification of the structure at the stage of molding products. In particular, a significant impact on the structure and properties of epoxy materials is their modification by ultrasonic treatment [1, 6]. Under the influence of ultrasound, the conditions of homogenization of the mixture, its viscosity, relaxation time and kinetics of structuring of epoxy composites change. Ultrasonic treatment allows increasing the deformation-strength and adhesion characteristics of epoxy composites, to reduce the residual stresses, to increase the durability and to significantly reduce the structuring time of the system.

In [7] it is shown that additional complex treatment of epoxy compositions at the stage of obtaining contributes to their structuring due to a more even distribution of structural components, activation of interface interaction and reduction defects of system. This allowed to increase the degree of structuring (G = 95.7 %) of the epoxy composite due to the formation of additional crosslinking sites between the active groups on the filler surface and the functional groups of the epoxy matrix.

In [8] the basic physical and chemical principles of formation of composite materials on the basis of epoxy resins are considered. The authors propose a new approach and substantiate the positive effect of complex ultrasonic treatment at the stage of formation of composite due to activation of the epoxy composition separately by ultrasonic. It is established that the use of this treatment with the subsequent introduction of the hardener in the epoxy oligomer increases the adhesion strength and cohesion strength, as well as the modulus of elasticity of the epoxy composite materials. However, the effect of ultrasound on the formation of epoxy composites in the case of treatment of a composition containing fillers has not been investigated.

In [9], the optimal mode of ultrasonic treatment was determined, in which the physical and mechanical and thermophysical properties of the anticorrosive epoxy composites, containing nanodispersed fillers, were increased. It is established that the optimal total duration of ultrasonic treatment of a composition with nanodispersed particles (q = 0.05 wt. %) is $\tau_t = (1.0 - 1.5) \pm 0.1$ min, and the pulse duration is $\tau_p = 10$ s. The total treatment time and the duration of the ultrasonic treatment of composition are the main factors that provide an increase in the adhesive and cohesive strength of the anticorrosive epoxy composites. However, the work did not investigate the structuring processes of epoxy composites, the compositions of which are processed ultrasound without a hardener.

In [10] investigated the effect of a low-frequency alternating magnetic field on the impact energy, heat resistance, modulus of elasticity, and flexural strength of the epoxy composite samples containing ferromagnetic filler. It has been found that the treatment with a low frequency alternating magnetic field of epoxy polymer compositions allows increasing by 4 – 5 % heat resistance of epoxy composites. The mechanical characteristics of epoxypolymers and epoxy composites are increased by 40-60 %. It is established that at the optimum processing time (60 min), the maximum effect is achieved at an alternating magnetic field frequency of 20 – 300 kHz. The paper determines that magnetic induction has the greatest influence on enhancing the cohesive characteristics of epoxy composites, the compositions of which were treated in a magnetic field. However, the influence of the magnetic field on the structuring of epoxy composites containing paramagnetic or diamagnetic fillers has not been investigated.

In [11, 12] it was found that the introduction of metal oxides powders contributes to the growth of the dynamic modulus of elasticity. The action of constant physical fields contributes to the reduction of the dynamic modulus of elasticity due to the ordering of the structure and the compaction of macromolecules of the matrix and filler particles in the epoxy composites. The use of external physical fields allows to change the plastic deformation capacity of epoxy composite materials from 5 % to 15 % and to reduce the degradation temperature by 50 K.

In [13], the positive effect of the treatment of compositions in physical fields on the increase in the resistance to cracking of epoxy composite coatings under the influence of the alternating temperature from -40 °C to +60 °C was confirmed. This treatment provides a uniform distribution of the filler in the composite material, improving the wetting of fine particles by surface activation. This improves the interphase interaction, reduces the residual stresses in the system, which greatly expands the temperature range of the use of the protective polymer composite coatings. However, the influence of physical fields on the structuring processes of epoxy composites filled with particles of different granulometric composition.

The purpose of this work is to optimize the composition of epoxy composites filled with fine powder of titanium oxide, the compositions of which are processed in physical fields at the stage of mixing components.

Research methods. Used epoxy resin ED-20 (GOST 10587-84), which is a high viscosity transparent liquid. The mass fraction of epoxy groups is 20.0 – 22.5 %. The mass fraction of volatile substances is 0.2...0.8 %. For curing epoxy polymer coatings polyethylenepolyamine – PEPA (TU 6-02-594-70) was used.

As a filler used fine powder of titanium oxide (TiO_2). Titanium oxide powder is prone to the formation of agglomerates in the air, which complicates the use of this filler. Agglomeration consists in the formation of new particles due to the aggregation of a certain number of primary particles. Agglomeration may result in partial or complete coalescence [14]. Aggregation increases the effective particle size, which is not a desirable phenomenon when forming a homogeneous structure of the composite.

Adhesive strength of epoxy composites was determined according to GOST 14759-69. The test material was applied to the end face of the tapered projection bars at the site of self-centering grips. The studies were performed on a breaking machine UMM-5 with a traverse speed of 2 mm/min.

The compressive strength was determined according to GOST 4651-82. Samples in the form of cylinders with a diameter of 10 ± 0.5 mm and a height of 15 mm were compressed with a traverse speed of 2 mm/min.

Impact energy was determined according to GOST 4647-80. Samples of rectangular shape with a square cross section of 10×10 mm and a length of 60 mm were subjected to dynamic loading on a pendulum impact machine with a charging angle of 160º.

The formation of the samples was to obtain a homogeneous composition, which included the necessary components. Depending on the samples volume, the quantitative content of the components in weight fraction is calculated at 100 wt. % of ED-20 epoxy resin. The formation of the epoxy composite material began with the dosing of the components, their stepwise introduction and mechanical mixing. To ensure a homogeneous structure of epoxy composites, their compositions were treatment by ultrasonic or magnetic field. The processing time was 5 min or 10 min. The formed composition was poured into special forms.

Research Results. It has been experimentally established that ultrasonic treatment of compositions with duration of 5 min increases the adhesive strength of epoxy composite materials (Fig. 1) filled with titanium oxide powder. The highest adhesive strength of 14.8 MPa was obtained for epoxy composite material containing 8 wt. % titanium oxide powder. The increase in the adhesion strength is due to the formation of a homogeneous system due to the intensive mixing of the components [12], which contributes to their even distribution in the volume of the composite. This results in better wetting of the filler particles due to the destruction of the agglomerates and the penetration of macromolecules of the epoxy polymer matrix into depressions, pores or cracks on the particles surface [7]. Ultrasonic treatment intensifies the degassing processes in the epoxy matrix, which contributes to the formation of defect-free products, especially when receiving high-filled systems with a high content of gas inclusions and high viscosity of the composition [13]. Removal of gas inclusions leads to an increase in the temperature of the system due to its heating by the absorbed energy of the physical field.

Ultrasonic radiation has little effect on the epoxy polymer system without fillers, which is confirmed by the low adhesive strength (11.3 MPa) for the epoxypolymer. This is due to the fact that the destruction of the adhesive layer occurs in the volume of the epoxypolymer. The cohesive strength of the epoxypolymer is lower than that of the filled system, the composition of which was processed with ultrasonic, which provided a better interaction between the components of the system. As the content of the filler increases, the adhesive strength increases, as the number inclusions of solid phase growing, which provides the formation of additional chemical bonds in the volume matrix.

It's investigated that increasing the duration of treatment to 10 min reduces the adhesive strength of epoxy composites by 11 – 15 %. This is because the prolonged ultrasonic influences intensify the structuring processes of the epoxy composite system, and, therefore, increase the residual stresses due to the uneven distribution of chemical bonds in the volume of the polymer matrix.

The fracture surfaces of adhesive layer of a low-filler epoxy composite (4 wt. %), the compositions of which were treated with ultrasonic for 5 min, contain coating fragments that are uniformly located on both parts of the face surface of the steel specimens (Fig. 2a). This indicates that the epoxy composites have a high adhesive strength, since this content ensures the formation of a homogeneous system. The level of residual stresses is low due to the uniform distribution of filler particles and the absence of stress concentrators at the phase interface.

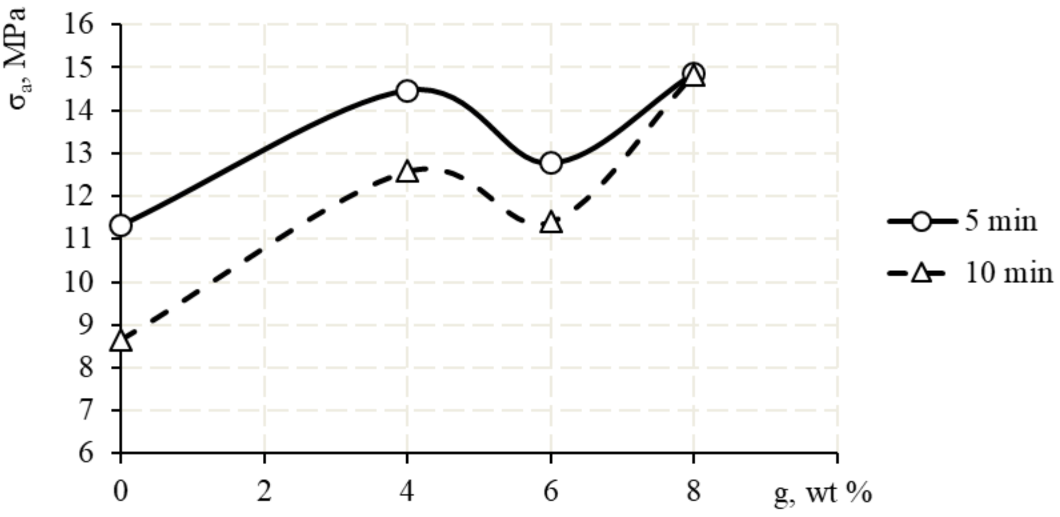

Fig. 1. The dependence of the adhesive strength of epoxy composites filled with titanium oxide powder from the mode of ultrasonic treatment of the composition

The surface of destruction of the adhesive layer of epoxy composites, the compositions of which were treated with ultrasonic for 10 min, occupies a much larger area of the epoxy composite coating on one face surface than on the other (Fig. 2b). This is due to the fact that the adhesive strength of epoxy composites is lower due to the excess processing time. Long-term ultrasonic treatment cause intensive structuring of epoxy composites, since the composition includes a hardener that begins to form chemical bonds with epoxy macromolecules. High-frequency ultrasonic vibrations cause the composition to heat up and increase the mobility of the macromolecule segments, resulting in the formation of chemical bonds at the concentration points of the macromolecule segments with the active groups. This type of structuring has a local and non-uniform nature, which leads to an increase in the level of residual stresses.

In the case of treatment of a composition, containing 8 wt. % of aluminum oxide powder, for 5 min the fracture surface of the adhesive layyr has a more pronounced adhesive nature, since on one face surface of the sample there is a larger part of the coating than on the other (Fig. 2, c). This means that the treatment time of 5 min is not sufficient to obtain a high degree of homogeneity of the system, since this composition has a higher content of fine filler, which is capable of forming agglomerates. These clusters of particles cannot be completely wetted by epoxy polymer binder, as a result, covalent bonds between each powder particle and the polymer matrix are not formed. The voids between the particles are defects in the polymer composite material and act as a source of microcrack formation.

The duration of ultrasonic treatment for 10 min of the composition containing 8 wt. % provides a more even distribution of the epoxy composite coating on the two face surface of the steel samples (Fig. 2, d). This is due to the fact that longer ultrasonic treatment of the composition causes the destruction of agglomerates and provides a more uniform distribution of disperse particles in the polymer matrix, increases the wettability of the particles by epoxy polymer binders, improves the interaction on the phase separation surface and promotes the formation of hydrogen bonds between the hydroxyl groups of the composition components.

In the case of low filler content (2 – 4 wt. %), no significant increase in strength of epoxy composites treated with ultrasonic (Fig. 3). This is due to the insufficient content of the particles that perform the reinforcing function. It should be noted that the ultrasonic treatment of the compositions for 10 min leads to a slight increase in this characteristic. Obviously, a longer ultrasonic treatment ensures the destruction of agglomerates and improves the interaction between the components. The compressive strength of epoxy composites, the compositions of which were treated with ultrasonic for 5 min, increases by 14-17 % in the case of the introduction 6-8 wt. % of titanium oxide powder compared to unfilled epoxy polymer and is 66.3 MPa and 68.5 MPa, respectively.

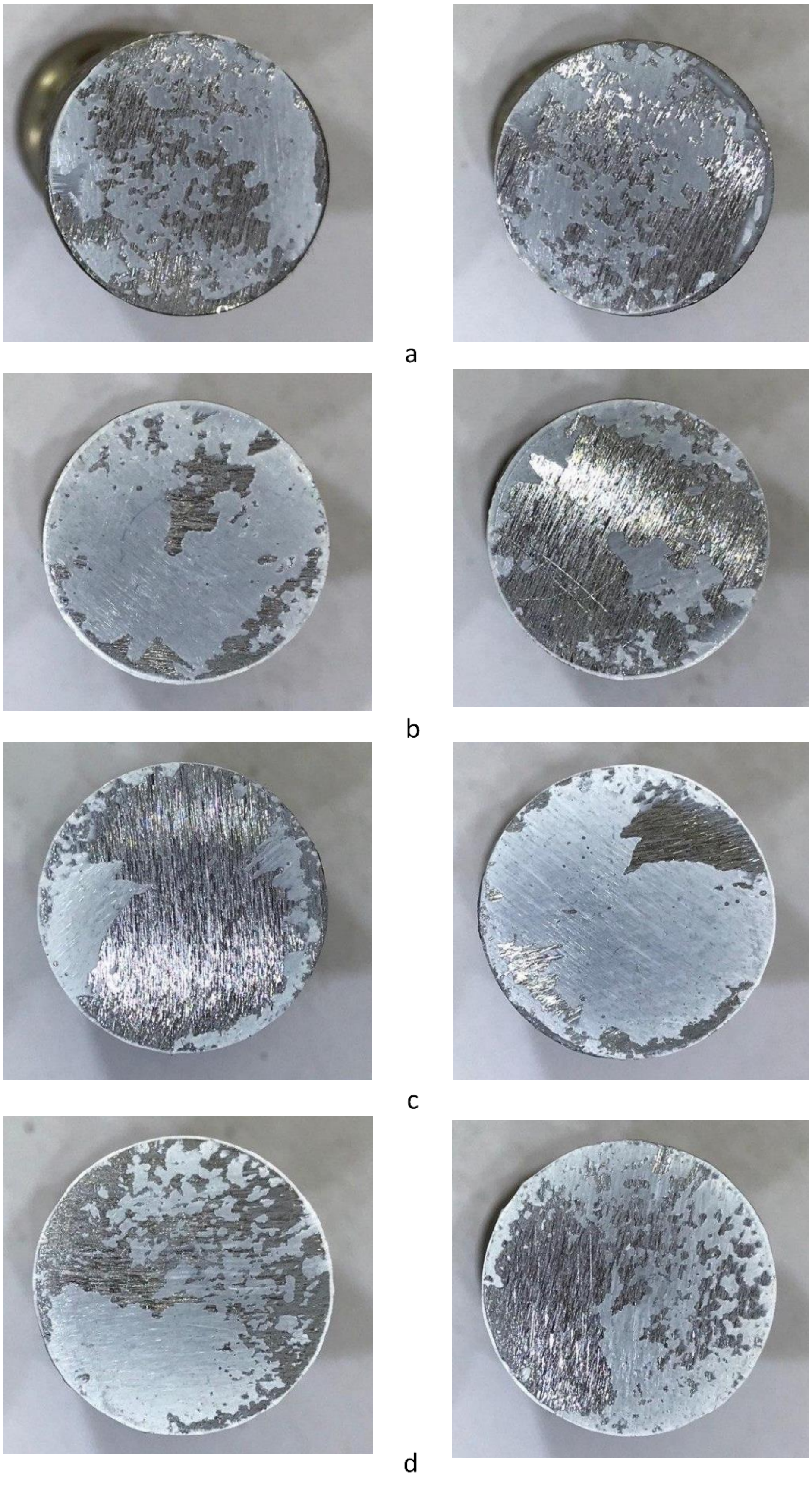

Fig. 2. View of the destroyed surface of glue connection of epoxy composites, filled with titanium oxide powder with ultrasonic treatment of composition:
a – 5 min, 4 wt. %; b – 10 min, 4 wt. %; c – 5 min, 8 wt. %; d – 10 min, 8 wt. %

Increasing the duration of ultrasonic treatment up to 10 min improve by 7 – 8 % compressive strength of epoxy composites containing 4 – 6 wt. % of powder. The low filler content is easier to distribute in the epoxy polymer matrix and to obtain a more uniform structure due to the influence of ultrasonic vibrations. However, low content does not provide a significant increase in strength, since the number of particles is insufficient to form a structure that can resist compressive loading.

Compared to studies on adhesive strength, a longer treatment of the compositions over 10 min reduces the strength of the epoxy composites in the case action of tensile loads. This is due to the fact that epoxy systems are more sensitive to fracture during stretching. Prolonged ultrasonic treatment results in residual stresses that cause cracks with smaller tensile loads. In the action of compressive loads, the presence of stress is not critical for the occurrence of cracks in the epoxy composite, since in this case, compensation for alternating stresses is provided, which increases the compressive strength in the case of treatment of the composition for 10 min.

Epoxy composites, the compositions of which were treated ultrasonic for 10 min, have the highest compressive strength (72.4 MPa). The introduction 8 wt. % of titanium oxide powder in the epoxy matrix provides increase in the compressive strength by 20 %. Increasing the time of ultrasonic treatment allows you to get more disperse particles, since high frequency oscillations during the optimum processing time are able to separate the particles from the formed agglomerates and provide a higher homogeneity of the epoxy system. Also in the process of ultrasonic treatment is the removal of the air inclusions from the composition and a less defective structure is formed that is capable of resisting higher compressive loading.

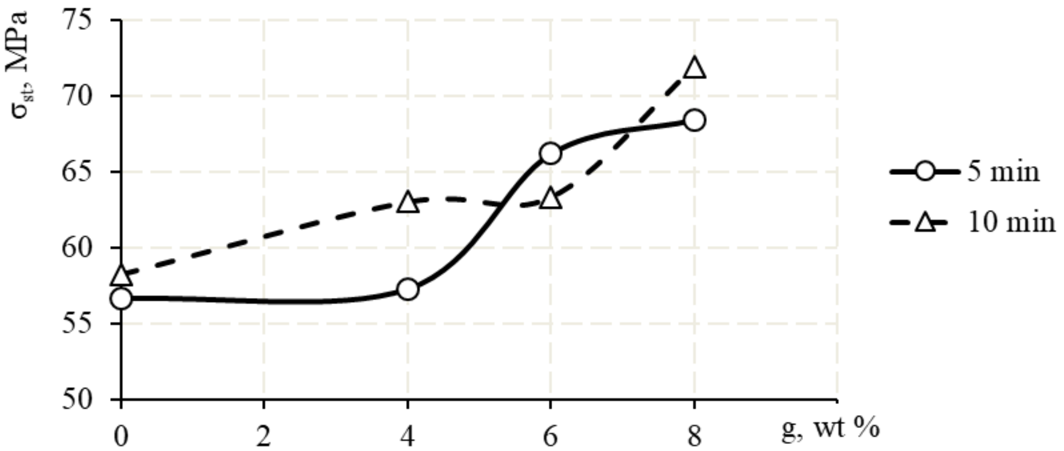

Fig. 3. The dependence of the compressive strength of epoxy composites filled with titanium oxide powder from the mode of ultrasonic treatment of the composition

For epoxy composites, compositions of which were treated ultrasonic, higher impact energy values were obtained compared to untreated epoxy composite materials [15]. The impact energy of unfilled epoxy composites, compositions of which were treated ultrasonic for 5 min, remained virtually unchanged (6.7 kJ/m^2) compared to the untreated materials (6.5 kJ/m^2). By increasing the duration of treatment of the compositions to 10 min there is a decrease of this characteristic by 20 %.

The impact energy of epoxy composites, the compositions of which are treated by ultrasonic for 5 min, increases by 22 – 25 % when the introduction 4 wt. % of titanium oxide powder compared to unfilled epoxypolymers (Fig. 4). The maximum value of impact energy is 8.6 kJ/m^2. This indicates the optimum content of the filler in the epoxy polymer matrix. Accordingly, after ultrasonic treatment, the particle aggregates of the filler are broken down, followed by their even distribution in the epoxy polymer matrix and formation of the structure of epoxy composite with the low stressed. The impact energy of epoxy composite materials containing 6 wt. % of titanium oxide powder decreases sharply 2 times compared to epoxy composites containing 4 wt. % of filler.

The lowest values of impact energy were obtained for epoxy composites with a filler content of 8 wt. %, that indicating the excess content of the filler in the epoxy polymer matrix. In this case, ultrasonic treatment for 5 min does not provide efficient separation of aggregates of filler particles, which increases the defective of structure of the epoxy composite. The results of the study of the impact energy of the low-filled epoxy composites (2 – 4 wt. %) correlate with the previous results of studies of adhesive strength and compressive strength, which confirms the positive impact of ultrasonic treatment at the optimal duration of 5 min.

The highest values of impact energy (5.3 kJ/m^2) have unfilled epoxypolymers compared to filled epoxy composites, the compositions of which were treatment ultrasonic for 10 min. Impact energy of epoxy composites containing 4 – 8 wt. % titanium oxide powder reduces, as there is local structuring, which leads to an increase in stress state of the system.

The content of titanium oxide powder in the amount of 6 wt. % provides a slight increase in impact energy by optimizing the structure, when there is dominance of the content of discrete particles, the number of which has increased due to ultrasound treatment, over the degree of the stress state of the epoxy system. The increased duration of ultrasonic treatment gives a positive result for epoxy composites that perceive compressive loads, leading to the destruction of particle agglomerates. However, for epoxy composites exposed to dynamic loads, the increased duration of ultrasonic treatment leads to rapid destruction of the material due to the growing in the residual stresses in the system.

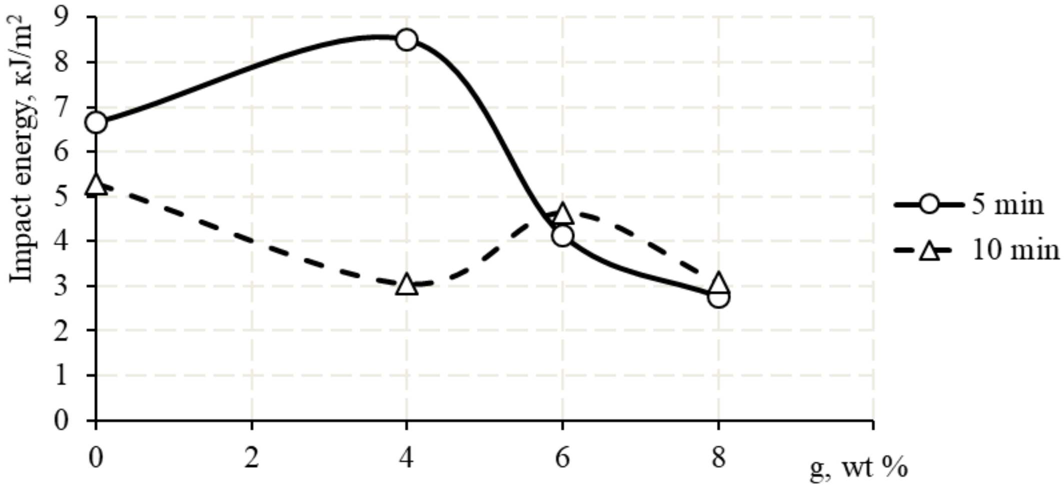

Fig. 4. The dependence of the impact strength of epoxy composites filled with titanium oxide powder from the mode of ultrasonic treatment of the composition

Conclusions. It is advisable to use ultrasonic treatment for epoxy compositions containing fine dispersed fillers, since high-frequency oscillations contribute to the destruction of particles agglomerates with high surface energy. In unfilled epoxypolymers, the macromolecule segments of the epoxy resin and hardener begin to oscillate under the influence of ultrasonic, which intensifies the formation of chemical bond. In the case of prolonged processing for more than 10 min, there is an increase in the residual stresses, which reduces the mechanical characteristics of epoxypolymers.

Increasing the duration of exposure to ultrasonic radiation up to 10 min is effective in the case of treatment of the composition containing of 6 – 8 wt. % of filler, which ensures the stability of the epoxy composite to plastic deformation and destruction under the influence of compressive loads. There is increase in the compressive strength of epoxy composites by 20 % due to the formation of additional bonds between the components of the system and the presence of a stronger phase compared to the epoxy polymer matrix. In this case, a long treatment (10 min) is necessary to convey sufficient energy for the composition. This ensures destruction of the agglomerates and the uniform distribution of the filler particles in the volume of the polymer matrix. Ultrasonic treatment reduces the degree of defective of structure of epoxy composites by removing the air inclusions that are trapped during mechanical mixing of the composition components.

The optimum content of titanium oxide powder is 4 wt. %, since the impact energy of epoxy composites, which are treated with ultrasound for 5 min, increase by 20 – 25 % compared to unfilled epoxy polymers. There is a uniform distribution of disperse particles in the epoxy polymer matrix and the formation of the structure of the epoxy composite with low stress state.

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