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Development of epoxy-polyester base modified with UV light for upgrading of technological equipment of vehicles

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Abstract

The paper studies the physical and mechanical properties, heat resistance of an epoxy-polyester composite material with the addition of methylene diphenyl diisocyanate modifier. The effect of ultraviolet radiation sources with wavelengths of 254 nm and 365 nm for different duration of treatment has been analyzed. It is found that the material that is exposed to ultraviolet irradiation for τ = 5 min at an irradiation wavelength of 365 nm is characterized by the optimal indicators of physical and mechanical properties.

The developed epoxy-polyester matrix is characterized by the following indicators of the properties under study: heat resistance (according to Martens) T = 354 K, breaking stresses during bending σ_b = 57.0 MPa, elastic modulus E = 3.7 GPa, impact strength W' = 12.6 kJ/m^2. For materials that have been treated with ultraviolet irradiation with a wavelength of 254 nm, the maximum values were obtained for the duration of treatment τ = 20 min. At the same time, there have been found low indicators of combined heat resistance, physical and mechanical properties in comparison with the composite treated with ultraviolet irradiation (365 nm): heat resistance (according to Martens) T = 350 K, breaking stresses during bending σ_b = 62.0 MPa, modulus of elasticity E = 3.5 GPa, impact strength W'= 11.8 kJ/m^2.

Additionally, the fracture surfaces of the developed materials were analyzed before and after preliminary treatment of the compositions with ultraviolet irradiation using optical microscopy. There have been revealed differences in the nature of the epoxy-polyester matrix fracture to irradiation and the matrix modified by ultraviolet light. It has been confirmed that the optimal conditions for ultraviolet irradiation of the studied composite material at a radiation wavelength of 254 nm is τ = 20 min; at a radiation wavelength of 365 nm τ = 5 min.

Keywords: *epoxy-polyester composite, flexural modulus, heat resistance (according to Martens), ultraviolet irradiation.*

Introduction

Today in various industries there are used polymer construction materials for various functional purposes [1–3]. The reason is that the elements of equipment based on them are characterized by an increased service life of the equipment, a decrease in the cost of parts and the mass of structures, and the like. One of the industries that is rapidly developing and requires new materials is the transport industry (river, sea, pipeline transport). An increase in the performance characteristics of technological equipment can be achieved by replacing metal parts with composite ones based on various polymer resins. An example of the use of polymer composite materials (PCM) is the protection of equipment surfaces from the effects of aggressive media, restoration of fan blades of gas cooling apparatus, sleeve bearings, hinges and fixed joints [4–6]. An increase in the performance characteristics of parts and mechanisms made on the basis of PCM can be achieved by introducing modifiers, fillers of various nature and dispersion into the polymer binder, changes in electric, magnetic fields, ultrasonic and ultraviolet processing of compositions. This makes it possible to increase the service life of vehicles and the economic efficiency of transportation in general.

The authors of [7, 8] have stated that it is promising to improve the adhesion and cohesive properties of composites based on an epoxy binder using the photochemical method of material modification. There was established presence of a "post-effect" after the termination of the action of ultraviolet (UV) exposure, which characterized both the surface and volumetric interaction of active radicals in the composition. At the same time, it has been investigated that polyester resins by their nature can be cured using ultraviolet radiation [8–10]. It is known that the treatment of materials with ultraviolet radiation is based on the chemistry of free radicals [11]. So, surface free radicals are formed by photodestruction of the surface by ultraviolet irradiation (surface activation). These processes occur on the surface of the material and affect only a few upper molecular layers (about 10^{-8} m). Under ultraviolet irradiation, electronically excited atoms and UV light attack the surface of the composition and

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destroy the C–C and C–H bonds, leaving radicals on the surface [11]. When free radicals are formed in this medium, they can only react with other radicals and, therefore, are stable. If there is any flexibility in the polymer chain, or if the radical moves along the chain, recombination, unsaturation, branching or crosslinking of free radicals can occur. Accordingly, it can improve the heat resistance and cohesive strength of the composite surface. However, after the introduction of the hardener into the composition, there is a movement of free radicals in the volume of the binder. Surface free radicals can interact with each other and penetrate into the bulk of the material, that is, there is a "post-effect", which is described in [12, 13]. It should be noted that the task of creating composite materials (CM) is multifactorial; therefore, all their properties should be taken into account when developing a polymer. Therefore, there is an urgent task of creating a polymer material that will differ in the complex not only in improved physical and mechanical, but also in thermophysical properties.

The aim of the work is to develop an epoxy-polyester matrix with improved physical, mechanical and thermophysical properties for the restoration of vehicles technological equipment.

Materials and research methods

When forming matrices for CM with improved adhesive, physical and mechanical properties, the following components have been used.

1. ED-20 epoxy oligomer (State Standard GOST 10587–84) (q = 100 parts by weight).

2. Orthophthalic dicyclopentadiene (DCPD) unsaturated before accelerated polyester resin ENYDYNE H 68372 TAE – q = 10 parts by weight (the content is indicated for 100 parts by weight of epoxy resin), which contains an inhibitor to prevent instant polymerization (gelation time τ = 20–24 min). It should be noted that during the reaction of copolymerization of unsaturated polyesters compositions with unsaturated monomeric compounds in the presence of initiators, a significant amount of heat is released, therefore the reaction is exothermic.

3. Cold hardener polyethylene polyamine (Pepa) (TU 6-05-241-202–78) with q=10 parts by weight (the content is indicated per 100 parts by weight of epoxy resin).

4. Initiator for polyester resins Butanox-M50 – q = 1.5 parts by weight, which is methyl ethyl ketone peroxide, and contains a low amount of water and a minimum amount of polar compounds compared to ethylene glycol.

5. Modifier of three-dimensional crosslinking methylene diphenyl diisocyanate, commonly known as pure MDI (4,4-MDI) q = 0.25 wt.h. Methylene diphenyl diisocyanate is an aromatic diisocyanate that is used for a three-dimensional crosslinking of polymers for polyurethane manufacturing. Chemical formula $CH_2(C_6H_4NCO)_2$, molar mass 250 g/mol, density 1180 kg/m^3.

The matrices were formed at a crosslinking temperature T = 393 ± 2 K. The concentration of the hardeners, 4,4-MDI modifier in the composition and the crosslinking temperature were established according to the preliminary results of the study.

In order to determine the optimal treatment mode for the epoxy-polyester binder with ultraviolet irradiation, the influence of the duration of treatment of UV sources with different radiation wavelengths was investigated. We used two UV sources with a central wavelength of 254 and 365 nm, respectively. The power of the lamps was W = 8V. The distance between the cuvette with the binder and the UV lamp was 40 cm. The duration of treatment varied within τ = 5–30 min.

Optimization of the processing mode was carried out according to the following properties of CM: heat resistance (according to Martens), flexural modulus, breaking stresses during bending, impact toughness.

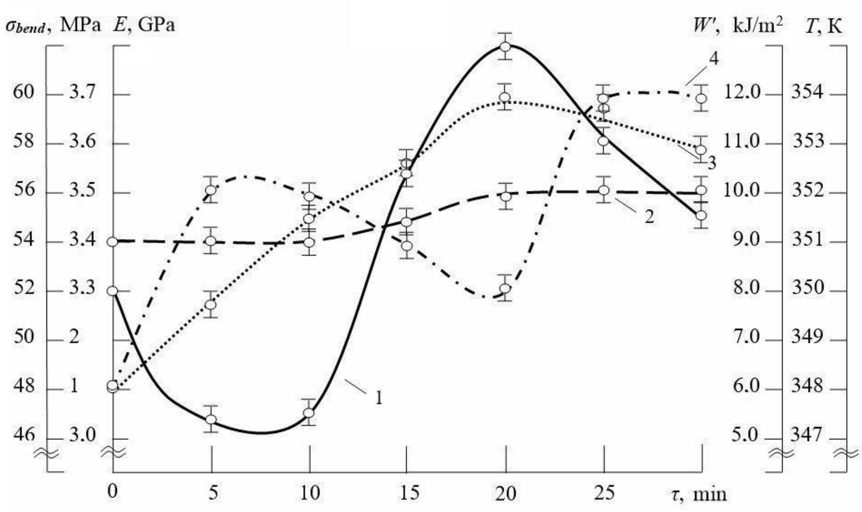
Heat resistance (according to Martens) of CM was determined in accordance with the State Standard GOST 21341–75. The research technique consisted in determining the temperature at which the test sample was heated at a rate of υ = 3 K/min under the action of a constant bending load F = 5 ± 0.5 MPa, as a result of which it was deformed by a given value (h = 6 mm).

Destructive stresses and flexural modulus were determined according to GOST 4648–71 and GOST 9550–81, respectively. Sample parameters were: length l = 120 ± 2 mm, width b = 15 ± 0.5 mm, height h = 10 ± 0.5 mm.

The impact strength was determined using a pendulum impact driver according to the Charpy impact method (GOST 4647–80). There was determined the working angle of deflection of the pendulum after the destruction of the sample at a predetermined initial angle of rise of the installation working body. The studies were carried out at temperature T = 298 ± 2 K and relative humidity d = 50 ± 5 %. Samples with the size: $l \times b \times h$ = (65 × 12 × 12) ± 0.5 mm were used.

Additionally, the structure of the CM fracture was investigated using optical microscopy. The studies were carried out using an XJL – 17AT series metallographic microscope equipped with a Levenhuk C310 NG Digital Camera (3.2 MegaPixels). The image magnification range is from ×100 to ×1600 times. During this work, the samples were examined at a magnification of 200 times. Levenhuk ToupView software was used to process digital images.

The materials were approved according to the experimentally established mode: the formation of samples and their holding for a time t = 12.0 ± 0.1 h at a temperature of T = 293 ± 2 K, heating at a rate of υ = 3 K/min to a temperature of T = 393 ± 2 K, holding the samples at a given temperature for a time t = 2.0 ± 0.05 h, slow cooling to a temperature of T = 293 ± 2 K. In order to stabilize the structural processes in the matrix, the samples were kept for a time t = 24 h in the air at a temperature of T = 293 ± 2 K, followed by experimental tests.



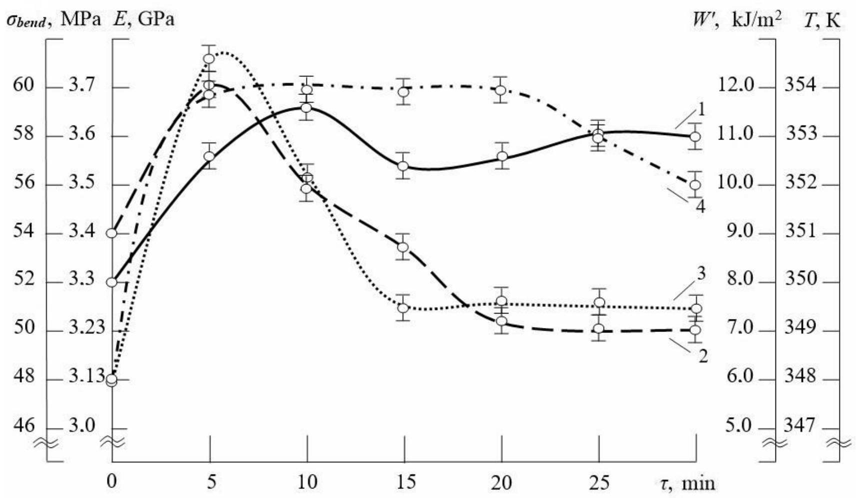
1 – breaking stress during bending (σ_b) 2 – flexural modulus of elasticity (E); 3 – impact strength (W′); 4 – heat resistance (T)

Figure 1 – Influence of UV exposure duration on the physical and mechanical properties and heat resistance of the 4,4-MDI matrix modified (254 nm)

Results and their discussion

At the stage of forming compositions, in order to increase the interfacial interaction of the components, there was carried out the physical modification of the epoxy-polyester binder using ultraviolet irradiation (before the introduction of fillers). To establish the optimal modes of the ultraviolet treatment process, the studies of the physical and mechanical properties of composite materials were carried out. At the first stage of experimental tests, there was established the effect of UV exposure on CM, which was modified using a bactericidal lamp DRB-8-1. The wavelength of ultraviolet rays was 254 nm. The duration of compositions irradiation (without a hardener) varied within τ = 5–30 min. It has been determined that, according to the duration of irradiation τ = 5 min, the indicators of destructive stresses during bending decrease from σ_b = 50.0 MPa (for a matrix modified 4,4-MDI) to σ_b = 47.5 MPa, the indices of the elastic modulus do not change (*E* = 3.4 GPa), and the impact strength increases from *W′*= 5.9 kJ/m^2 to *W′* = 7.8 kJ/m^2 (Fig. 1, curves 1–3). It should be noted that with this duration of UV exposure, heat resistance indices increased from *T* = 348 K to *T* = 352 K (Fig. 1, curve 4). With an increase in the processing time up to τ = 10 min, the investigated properties (destructive stresses during bending, elastic modulus and heat resistance) do not change (Fig. 1, curves 1, 2, 4). In particular, there was found an increase in impact strength indicators by $\Delta W'$= 1.6 kJ/m^2. A further increase in the duration of UV exposure of compositions by τ = 20 min provides an increase in the indicators of physical and mechanical properties. The indicators of breaking stresses increase from σ_b = 47.5 MPa to σ_b = 62.0 MPa, the modulus of elasticity – from *E* = 3.4 GPa to *E* = 3.5 GPa and impact strength to *W′*= 11.8 kJ/m^2. It should be noted that the heat resistance of such materials decreases to *T* = 350 K. With an increase in the UV exposure to τ = 30 min, there is observed a decrease in the physical and mechanical properties of CM: breaking stresses σ_b = 55.0 MPa, elastic modulus *E* = 3.5 GPa and impact strength W'= 10.9 kJ/m^2. At the same time, the indicators of heat resistance (according to Martens) grow to *T* = 354 K (the results are within the experimental error). The decrease in the indices of physical and mechanical properties at τ = 30 min can be explained by the effect of degradation of the epoxy-polyester binder due to an increase in the duration of UV exposure [14].

Figure 2 shows the results of a study of the effect of ultraviolet exposure (with a radiation wavelength of 365 nm) on the investigated properties. The duration of this exposure has remained unchanged: τ = 5–30 min. It has been experimentally established that the indicators of physical and mechanical properties and heat resistance increase with the duration of ultraviolet irradiation τ = 5 min: Indicators of breaking stresses increase from σ_b = 50.0 MPa (for a matrix modified 4,4-MDI) to σ_b = 57.0 MPa , elastic modulus indices – from *E* = 3.4 GPa to *E* = 3.7 GPa, and impact toughness – from *W′*= 5.9 kJ/m^2 to W' = 12.6 kJ/m^2 (Fig. 2, curves 1–3). In this case, the heat resistance increases from *T* = 348 K to *T* = 354 K. With an increase in the duration of UV exposure to τ = 10 min, there was observed a slight decrease in the parameters of the modulus of elasticity and impact toughness: *E* = 3.5 GPa, *W′*= 10.2 kJ/m^2. It should be noted that the indicators of breaking stresses during bending increased by $\Delta\sigma_b$ = 2.1 MPa, and the heat resistance did not change (Fig. 2, curve 4). With a further increase in the duration of UV exposure to τ = 20–30 min, there was noted a decrease in the elastic properties and resistance of the investigated materials to shock loads. So, the modulus of elasticity in bending decreased to *E* = 3.2 GPa, and the impact strength decreased to *W′*= 7.5–7.7 kJ/m^2. The indices of breaking stresses during bending and heat resistance according to Martens with such a treatment duration varied within the



1 – breaking stress during bending (σ_b); 2 – flexural modulus of elasticity (E); 3 – impact strength (W'); 4 – heat resistance (T)

Figure 2 – Influence of UV exposure duration on physical and mechanical properties and heat resistance of the modified MDI matrix (365 nm)

experimental error (σ_b = 57.0–58.0 MPa, T = 352–354 K) (Fig. 2, curves 1, 4).

Analyzing the results of experimental studies, it was determined that under UV exposure with a radiation wavelength of 254 nm, the maximum indicators of physical and mechanical properties characterized the material according to the processing time τ = 20 min: breaking stresses in bending σ_b = 62.0 MPa, elastic modulus E = 3 5 GPa, impact strength W'= 11.8 kJ/m^2. It was found that the heat resistance of materials decreased to T = 350 K. At the same time, under UV exposure with a radiation wavelength of 365 nm, the material is characterized by the maximum processing time τ = 5 min. The following values of the properties studied were obtained: breaking stresses in bending σ_b = 57.0 MPa, elastic modulus E = 3.7 GPa, impact strength W'= 12.6 kJ/m^2 and heat resistance (according to Martens) is T = 354 K. Thus, it has been established that, from a practical and economic point of view, it is advisable to modify the UV exposure matrix with a radiation wavelength of 365 nm according to the processing time τ = 5 min.

The obtained results of experimental studies can be explained by the course of physicochemical processes in epoxy-polyester compositions under UV exposure and the formation of additional bonds of the 4,4-MDI modifier with the binder. It is known that highly reactive modifier groups (OH/NCO) can react with carboxyl and hydroxyl groups of an epoxy-polyester binder [15].

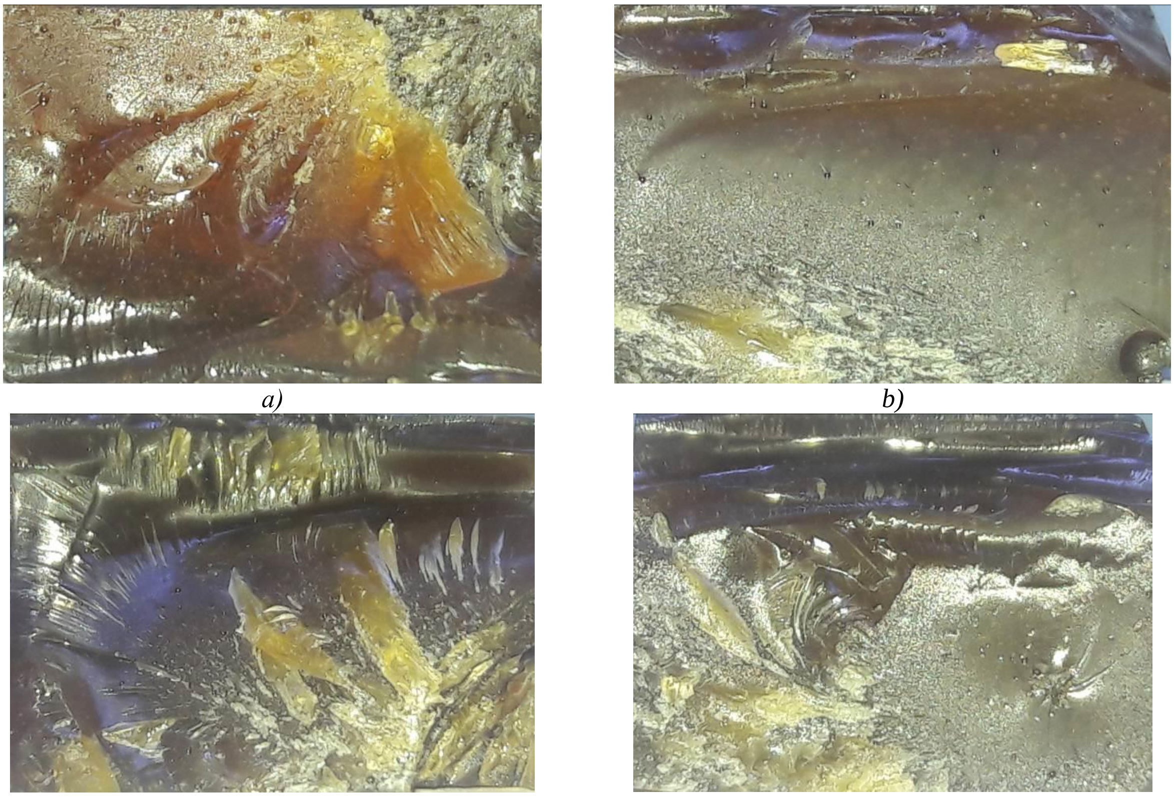
At the next stage, the fracture surfaces of composite materials were qualitatively investigated using optical microscopy. Figure 3 shows the fracture surfaces of materials that were treated with ultraviolet exposure with a wavelength of 254 nm for a duration of τ = 0–30 min. A qualitative analysis of the photographs made it possible to confirm a decrease in the breaking stresses during bending at the duration of UV exposure τ = 10 min (Fig. 3, *b*). The surface of such materials is characterized by straight fracture lines and the presence of stress concentrators, which leads to a decrease in properties. The analysis results of surfaces of the samples fracture according to the duration of UV exposure τ = 20–30 min completely correlate with the obtained values of the physical and mechanical properties. According to Fig. 3, *c, d* it follows that such materials are characterized by a viscous fracture surface and the presence of chaotic shear lines. This indicates an increase in the cohesive bonds of materials after ultraviolet exposure for a given duration of treatment.

Figure 4 shows the fracture surfaces of epoxy-polyester materials after ultraviolet exposure for τ = 0–30 min with a radiation wavelength of 365 nm. According to the processing time τ = 5–10 min, it was found a more chaotic grid of surface fracture (in comparison with the matrix, Fig. 3, *a*). This confirms the dependence of the cohesive properties on the duration of UV exposure. So, with an increase in the indicators of physical and mechanical properties, there is observed multidirectional propagation of cracks when the material is fractured, characterizing such a material as less brittle. A decrease in the randomness of the propagation of a fracture occurs due to an increase in the duration of ultraviolet irradiation τ = 20–30 min (Fig. 4, *c, d*). The level of breaking of such materials should have a smooth fracture structure, and the destruction of the samples took place in the same plane.

It should be noted that the optimal conditions for UV exposure determined at the first stage (τ = 20 min, 254 nm; τ = 5 min, 365 nm) fully correlate with the analysis results of the fracture surfaces of the studied samples.

Conclusions

The experimental studies have established the effectiveness of pretreatment of an epoxy-polyester binder modified with 4,4-MDI, ultraviolet rays of different wavelengths.

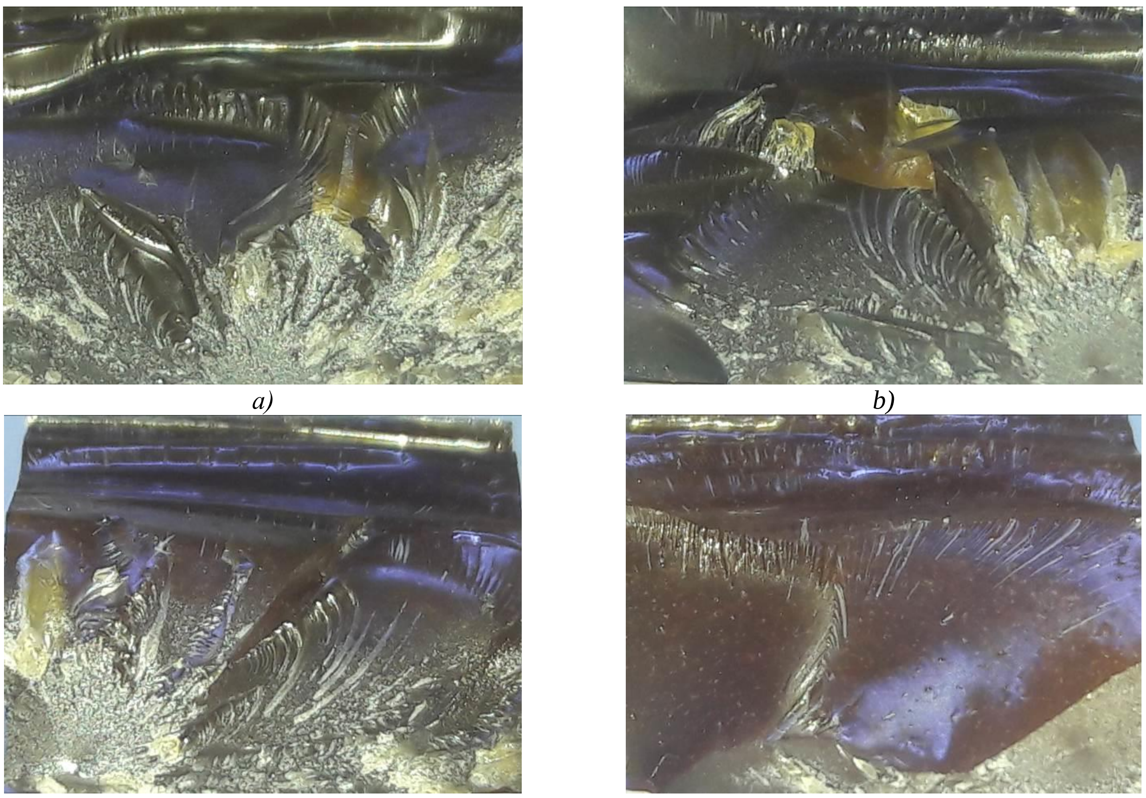


a) *b)*

c) *d)*

a) τ = 0 min; b) τ = 10 min; c) τ = 20 min; d) τ = 30 min

Figure 3 – Typical fracture surfaces of epoxy-polyester materials after UV exposure (254 nm) with different duration of treatment: a) matrix (control sample)



a) *b)*

c) *d)*

a) τ = 5 min; b) τ = 10 min; c) τ = 20 min; d) τ = 30 min

Figure 4 – Typical fracture surfaces of epoxy-polyester materials after UV exposure (365 nm) with different duration of treatment

An increase in physical and mechanical properties and heat resistance (according to Martens) with a radiation wavelength of 254 nm with a processing time of τ = 20 min has been established. It has been determined that the indicators of breaking stresses during bending increase from σ_b = 50.0 MPa (for a matrix modified by 4.4-MDI) to σ_b = 62.0 MPa, the indices of the elastic modulus increase from E = 3.4 GPa to E = 3.5 GPa, the impact strength increased from W'= 5.9 kJ/m^2 to W' = 11.8 kJ/m^2. In this case, the heat resistance increased from T = 348 K to T = 350 K.

It has been stated that the maximum indicators of the properties under study are characteristic of the composite material under ultraviolet irradiation for τ = 5 min (the radiation wavelength is 365 nm). The following values of indicators of physical and mechanical properties have been determined: breaking stresses in bending σ_b = 57.0 MPa, elastic modulus E = 3.7 GPa, impact strength W'= 12.6 kJ/m^2 and heat resistance (according to Martens) is T = 354 K.

There has been carried out a qualitative analysis of the fracture surfaces of composite materials before and after preliminary treatment of the compositions with ultraviolet exposure. Differences in the character of sample cracking after UV exposure (in comparison with the epoxy-polyester matrix without this exposure) have been revealed. It has been confirmed that the optimal conditions for UV exposure of the investigated composite materials at a wavelength of 254 nm are τ = 20 min; at a wavelength of 365 nm τ = 5 min. The results obtained make it possible to state the possibility of using the developed material for the restoration of technological equipment elements of vehicles.

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Розробка епокси-поліефірної основи, модифікованої ультрафіолетовим опромінення, для відновлення технологічного устаткування транспортних засобів

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У роботі досліджено фізико-механічні властивості та теплостійкість епокси-поліефірного композитного матеріалу при додаванні модифікатора метилендіфенілдіізоціанату. Проаналізовано вплив джерел ультрафіолетового опромінення з довжинами хвиль 254 нм та 365 нм за різної тривалості обробки. Встановлено, що оптимальними показниками фізико-механічних властивостей характеризується матеріал, який піддавали ультрафіолетовому опроміненню упродовж $\tau = 5$ хв за довжини хвилі опромінення 365 нм. Розроблена епокси-поліефірна матриця характеризується наступними показниками досліджуваних властивостей: теплостійкість (за Мартенсом) T = 354 K, руйнівні напруження при згинанні $\sigma_{зг} = 57.0$ МПа, модуль пружності E = 3.7 ГПа, ударна в'язкість W' = 12.6 кДж/м^2. Для матеріалів, які обробляли ультрафіолетовим опроміненням з довжиною хвилі 254 нм отримали максимальні значення за тривалості обробки $\tau = 20$ хв. При цьому встановлено у комплексі нижчі показники теплостійкості і фізико-механічних властивостей, порівняно з композитом обробленим ультрафіолетовим опроміненням (365 нм): теплостійкість (за Мартенсом) T = 350 K, руйнівні напруження при згинанні $\sigma_{зг} = 62.0$ МПа, модуль пружності E = 3.5 ГПа, ударна в'язкість W' = 11.8 кДж/м^2. Додатково проаналізовано поверхні зламу розроблених матеріалів до та після попередньої обробки композицій ультрафіолетовим опроміненням за допомогою оптичної мікроскопії. Виявлено відмінності у характері зламу епокси-поліефірної матриці до опромінення та матриці, модифікованої ультрафіолетом. Підтверджено, що оптимальні умови ультрафіолетового опромінення досліджуваного композитного матеріалу за довжини хвилі випромінювання 254 нм $\tau = 20$ хв; за довжини хвилі випромінювання 365 нм $\tau = 5$ хв.

Ключові слова: *епокси-поліефірний композит, теплостійкість (за Мартенсом), ударна в'язкість, ультрафіолетове опромінювання.*