INVESTIGATION OF THE IMPACT STRENGTH OF THE EPOXY-POLYESTER MATERIAL FOR USING IN AGGRESSIVE ENVIRONMENTS IN THE WATER TRANSPORT

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Resume

The influence of the aggressive environments of river water, seawater, oil, petrol and sodium hydroxide on the impact strength of epoxy-polyester composite was investigated. Testing was conducted by two different methods. It was determined that the impact strength of specimens that were kept at air was $W' = 4.2$ kJ/m^2 (by the second method - $W'' = 7.0$ kJ/m^2). The energy spent on destruction of the control specimen was $E = 0.92$ J. The main regularities of the crack propagation process in the studied material were found and qualitatively described. The time of a crack propagation (= 0.16 μs) was determined. The maximum load, which leads to destruction of the specimen was $P_{max} = 2.47$ kN. The comparative analysis of investigated specimens was conducted by the IR-spectroscopy method. It was found, that the biggest decrease in material properties was observed for the specimen that was kept in the petrol environment.

1   Introduction

The technical readiness of a vessel to the accomplishment of its functions, reliability and economics of works, directly depends on its support in the satisfactory technical condition. The construction elements of a hull, mechanical plants, mechanisms, devices and systems are constantly exposed to the static and dynamic load. The vessel is a complex technical construction of high danger, all the life cycle of which passes in unfavourable conditions of operation (aggressive water environment, conditions of elevated temperatures, alternate and pulsed loads, excessive friction, trimming moments and etc.) [1]. Sea and river vessels are strategic industrial objects and, therefore, the ship owners put in claims of high demands for their long-term and reliable exploitation. Protective polymer coatings are used to protect the metal surfaces of parts and mechanisms of the vessel [2-6]. At the same time, the use of parts based on polymer composite materials (PCM) can significantly reduce their costs and overhaul time [7]. Such materials are characterized by increased physical and mechanical, thermophysical properties and wear resistance, which allow increasing of a service life of parts and mechanisms. In addition, it does not affect the increase in the costs of the vessel's repairing. Taking into account the influence of the aggressive environment

on the working parts of such transport vehicles is important in this case. As it is known that physically and chemically active aggressive environments can have the same negative effect on the strength of composite materials (CM) and coatings on their basis (change of stiffness, cracking and complete destruction), but have different mechanism of influence [8]. Therefore, the development of the CM, resistant to impact of aggressive environments, is an actual practical task.

Based on previous studies of the polymer strength, it was found that the mechanism of the specimen destruction is associated with the process of growth and propagation of microcracks [9]. In accordance with the statistical theory, the fragile destruction of the specimen occurs gradually and not simultaneously by the entire surface of destruction. In this case, development of the destruction process of the specimen is investigated as a function of time. The fracture destruction is considered to be an irreversible time process of accumulation of structural damage in the volume of a deformed specimen [9]. The work, consumed for the specimen fracture, is one of the characteristics by which it is possible to estimate the strength of the material. It is attributed to the unit of the destruction surface (impact strength) [10]. The durability of the composite and the protective coating based on it, the resistance to statical and dynamical loading, can be estimated by its value [11]. In the works

[12-13], the impact strength of the CM of the different nature was investigated. Effective regimes of their forming and causes of structural degradation were determined. Processes of localization of deformations and destruction of the CM were investigated. The shock characteristics of the CM, modified by the energy and magnetic fields, ultraviolet irradiation, by addition of fillers of varying dispersion and nature were investigated by authors [14]. Literature analysis showed that most studies were conducted under normal conditions (pressure, temperature, humidity). At the same time, the study of the physically and chemically active aggressive environments influence on the shock properties of CM is insufficiently studied. Obtained results would allow predicting the behavior of the developed material in real conditions.

2 Materials and methods

The following components were used for the formation of the CM matrix:

a) Epoxy resin ED-20 ($q = 100$ wt%).

b) Orthophthalic dicyclopentadiene (DCPD) unsaturated pre-accelerated polyester resin ENYDYNE H 68372 TAE - $q = 10$ wt% (hereinafter are given per 100 weight parts of epoxy resin), which has an inhibitor to prevent instant polymerization (gel time from $t = 20$ to 24 min). It should be noted that, during the copolymerization reaction of the composition of unsaturated polyesters, with unsaturated monomer compounds in the presence of initiators, a significant amount of heat is released, therefore the reaction is exothermic.

c) The cold curing hardener polyethylene polyamine (PEPA) - $q = 10$ wt%.

d) The initiator for polyester resins Butanox-M50 - $q = 1.5$ mas.fr (the content is indicated on 100 weight parts of polyester resin), which is methyl ethyl ketone peroxide (MEKP) and contains a low amount of water and a minimum amount of polar compounds in comparison to ethylene glycol.

e) Modifier methylene diphenyl diisocyanate, widely known as MDI (4,4-MDI) - $q = 0.25$ wt%. Methylene diphenyl diisocyanate is aromatic diisocyanate, which is used for the three-dimensional cross-linking of polymers in the manufacture of polyurethane. Chemical formula: $CH_2(C_6H_4NCO)_2$, molar mass - 250 g/mol, density - 1180 kg/m³.

f) Filler 1: Hexagonal boron nitride (h-BN) - 8-10 µm - $q = 60$ wt%

g) Filler 2: Mica 20-40 µm - $q = 20$ wt% (The fractionated micromica of series "Standard" (ISO 3262-12:2001, Extenders for Paints. Specifications and Methods of Test. Part 12. Muscovite-Type Mica) of the brand MC-20-80 with grains d = 20 - 40 µm. Mineral composition: mica-muscovite KAl_2 [(OH, F)$_2$AlSi$_3$O$_{10}$]. Chemical composition according to ISO 3262-12:2001: silicon oxide (SiO_2) - 44-50%, aluminium oxide (Al_2O_3) - 27-35%, iron oxide (Fe_2O_3) - less than 5%, magnesium oxide (MgO) - less than 2%, potassium oxide (K_2O) - more than 8%, water-soluble salts - less than 0.2%).

The composition without hardeners was modified by the ultraviolet irradiation for duration of 5 min before introducing of hardeners.

The specimens were solidified according to the following regime: the formation of specimens and their holding over time t = 12.0±0.1 h at a temperature T = 293 ± 2 K, heating at a rate of υ = 3 K/min to the temperature of T = 393 ± 2 K, keeping the specimens at a given temperature during the time t = 2.0 ± 0.05 h, slow cooling to the temperature of T = 293±2 K. For stabilization of the structural processes in the matrix, the specimens were kept during the time t = 24 h at air at the temperature of T = 293 ± 2 K, with subsequent experimental tests. Further, the formed specimens were kept in the following environments: oil, seawater, river water, petrol, sodium hydroxide (NaOH (50%)) during the 30 days. The control specimens were kept at air during that time.

The impact strength, relative mass change of specimens and activity of the surface of composite material were investigated by the IR-spectral analysis method.

The impact strength was determined by two methods.

The first method (W'). The impact strength was measured by pendulum copra according to Charpy method (ASTM D6110). The working angle of deviation of the pendulum after the destruction of the specimen, at a predefined initial corner of lifting of working body of installation, was determined. The investigation was conducted under the temperature of $T = 298 \pm 2$ K and relative humidity of $d = 50 \pm 5\%$. The specimen dimensions were: $l \times b \times h = (63.5 \times 12.7 \times 12.7) \pm 0.5$ mm.

The second method (W''). The impact strength was measured with use of the notched specimens of sizes $10 \times 15 \times 75$ mm (ASTM D6110). The tests were carried out with use of the impact pendulum machine RKP-300 under the high-rate loading (5.2 m/s). In doing so, the loading diagrams, "load-time" and "load-deflection", were registered. A total work of impact fracture E is composed of two components: i) crack initiation E_i and ii) crack propagation E_p.

The impact strength was determined as:

$$W = \frac{E}{bs}, \tag{1}$$

where E is the impact energy, consumed for the specimen fracture; b is the width of a specimen; s is the thickness of a specimen.

The impact loading diagram processing software "VUHI-CHARPY" was used to determine the fracture energy components by transforming the "loading - time" (P-t) dependence into the "loading - displacement" (P-s).
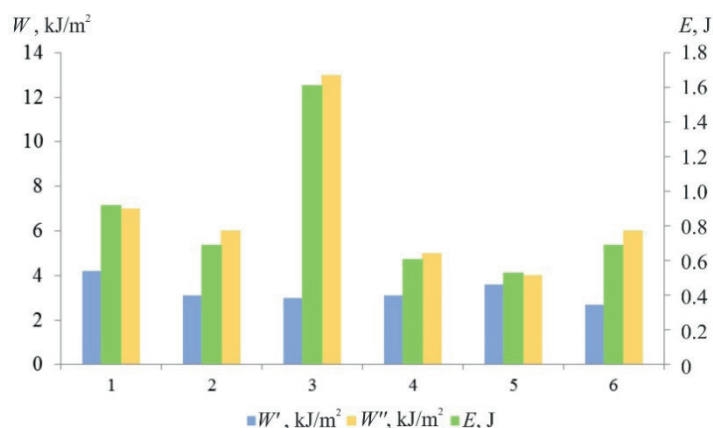
Figure 1 *Influence of aggressive environments on the impact strength (W' and W")*
and energy (E) spent on the destruction of material: 1) air (control specimen);
2) oil; 3) seawater; 4) river water; 5) petrol; 6) NaOH (50%)

The mass of specimens was determined by weighing with analytical scales, with an accuracy of ± 0.001 g.

Deviation of values of impact strength and mass change was 4 - 6 % from nominal, during the study.

The chemical bonds on the composite surface were investigated with use of the IR-spectral analysis. The IR-spectra were registered with "IRAffinity-1" (Japan) spectrophotometer at the wave numbers range from v = 400 to 4000 cm^{-1} by a single-beam method in the reflected light. The wavelength scanning at wave number $\lambda^{-1} = v$ was performed at diagrams within 225 mm, in the range of the above specified frequencies. The wave numbers (v, cm^{-1}), absorption band intensity (T, %) and absorption band area (S, %) were determined by a computer program "IRsolution". The error of the wave number measurement was v = ± 0.01 cm^{-1}, while the error of the peak location made v = ± 0.125 cm^{-1}. The photometrical accuracy was ± 0.2% at software operated control of cracks and integration time t = 10 s. The integration step was Δv = 4 cm^{-1}.

3   Results and discussion

At the first stage, the investigation of the impact strength of the material by two different methods was conducted after keeping specimens in different aggressive environments during the 30 days. The influence of physically and chemically active aggressive environments on the CM strength was studied. River water, seawater, oil environment, petrol were attributed as the physically active aggressive media and sodium hydroxide (50%) - as the chemically active (according to the classification of Zuyev, [9]). An influence of aggressive environments on the stability of composite materials to shock loads was analyzed. The investigation results are shown in Figure 1. It was found, that the impact strength of the control specimens was W' = 4.2 kJ/m^2 by the first method and W'' = 7.0 kJ/m^2 - by the second method. It should be noted, that the energy spent on the destruction of the control specimen

was E = 0.92 J. Analysis of the experimental results allowed to determine the insignificant decrease of the impact strength of the specimens, which were kept in the aggressive environments (oil, seawater, river water). The impact strength values were W' = 3.0 - 3.1 kJ/m^2 (by the second method - W''= 5.0 - 6.0 kJ/m^2), except of for specimens, which were kept in seawater (by the second method W'' = 13.0 kJ/m^2). Hence, the energy, spent on destruction of specimens, decreased to the E = 0.61 - 0.69 J and for the specimen that was kept in seawater, the energy increased to E = 1.6 J. The impact strength of the specimens, which were kept in petrol was W' = 3.6 kJ/m^2 (by the second method - W'' = 4.0 kJ/m^2). Moreover, the energy, spent on destruction of the CM, decreased to E = 0.53 J (compared to the control specimens) (Figure 1). The impact strength of specimens that were kept in the alkaline environment, by the first method was - W' = 2.7 kJ/m^2 (by the second method - W''= 6.0 kJ/m^2). The energy spent on destruction of the specimen was E = 0.69 J.

In addition, it is important to determine the force that initiated the crack growth, since, at a shock load, the wave of stress passes through the specimen and destruction occurs when the critical stress is reached [15]. In turn, it would allow to determine the resistance to destruction of polymer materials and to evaluate the initiation and propagation of cracks after the impact loading. According to the second method, the investigation of the "load-time" (Figure 2, a) and "load-deflection" (Figure 2, b) dependences were carried out. During the analysis of the experimental data (Figure 2, Table 1), it was determined that the destruction of polymeric materials in a shock load occurs in several stages. It is known that behavior of the polymers on fatigue cracks is usually divided in three discrete phases: first - the threshold phase indicating the initiation of the crack (I); second - the phase of stable crack propagation (II); third - the phase of fast and unstable growth of cracks (III) (Figure 2, a) [16]. Analogous phases of the crack growth are indicated in works [14, 17], which allows to assert about the reliability of the obtained results.

Table 1 *The results of experimental investigation of polymer materials, which were kept in different environments*

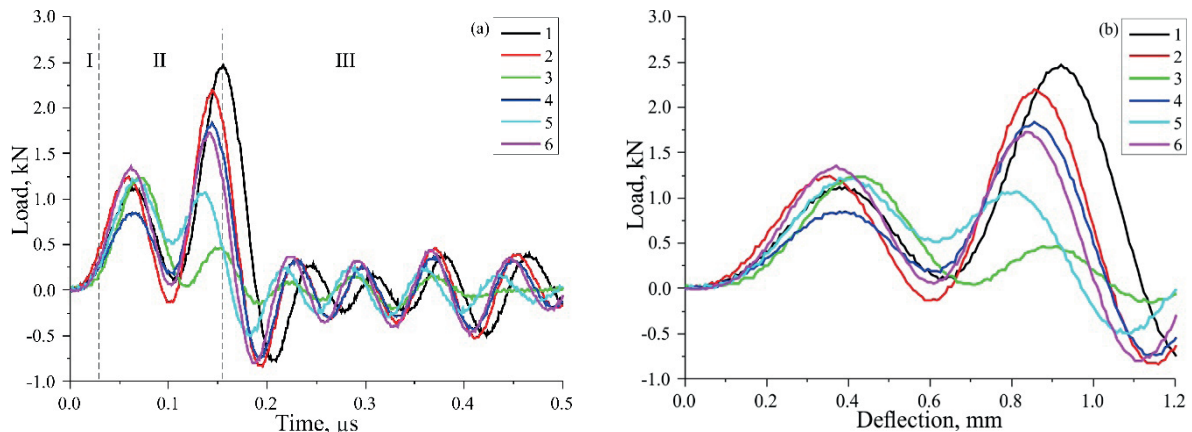| environment	load at which initiated crack propagation, P_{max} (kN)	time of crack propagation, τ (µs)	the value of critical deformation, Δl (mm)
air	2.47	0.16	0.92
oil	2.20	0.14	0.86
seawater	1.24	0.07	0.44
river water	1.84	0.14	0.86
petrol	1.22	0.07	0.41
NaOH (50%)	1.73	0.14	0.84

Figure 2 *Dependence of load on time (a) and deflection (b) of specimens, which were investigated in different environments: 1) air; 2) oil; 3) seawater; 4) river water; 5) petrol; 6) NaOH (50%)*

It was found, that the curves' shapes of the "load-time" at the beginning of the main crack growth (phases I and II, Figure 2a) are almost identical. This confirms that the initiation and growth of the crack for all specimens occurs at analogous phases. The load-deflection curves for all the specimens have a linear dependence with a sudden drop in load to zero at the time of initiation of the crack growth. Two peaks were found at the regime of the stable crack propagation. It was due to the "structural reorganization" of the specimens' material [18-19]. The time of macrocrack growth of the control specimen was $\tau = 0.16$ µs (Figure 2 a, Table 1). At the same time, the maximal load was $P_{max} = 2.47$ kN. The critical deformation at this load was $\Delta l = 0.92$ mm (Figure 2 b). The expense of energy on the main cracks propagation for the specimens, which were kept in oil, river water and alkaline reached the maximum values at the same time interval $\tau = 0 - 0.14$ µs. The crack growth initiation occurred with a slight decrease in load to $P_{max} = 1.73 - 2.20$ kN. At the same time, the value of the critical deformation for those materials was determined - $\Delta l = 0.84 - 0.86$ mm. Reducing resistance to the crack growth under shock loads is associated with a decrease in energy spending, which is characterized by the dispersive brittleness of the material. This is accompanied by a decrease in the maximum load at which the main crack arises.

In addition, at the second phase, the shift of the stress intensity range (in the direction of decrease) required for the initiation of the crack growth of specimens 3 and 5, was observed (Figure 2 a). Therefore, propagation of the main crack to reaching the maximum load occurred at: $\tau = 0.07$ µs (Figure 2 a, Table 1). At the same time, the shape of curves 3 and 5 is typical for ductile failure throughout the studied range [20]. Taking into account this fact, it can be stated, that influence of aggressive environments on the impact properties of the developed materials is of a very slight degree.

At the stage of unstable cracks growth (Figure 2 a, III), processes of oscillations of small amplitude were observed. They are related to appearance of stripes of localization of deformations. These processes are associated with the heterogeneity of the material structure, the heterogeneous stress-strain state and the dynamic excitation of deformation processes due to the diffusion of the impact energy. The specimen material ceases to resist the crack propagation. Evidently, at this stage, the main crack increases until the complete destruction of the specimen. During the experimental data analysis, the time of the complete destruction of the specimens was determined (τ'). For the whole series of experiments, a time range of fracture was - $\tau' = 9.46 - 9.73$ µs. This indirectly indicates an insignificant influence of the aggressive environment on the impact properties of the developed epoxy-polyester composite.

Study of the mass changes of specimens after the testing was performed in order to estimate the resistance of the developed CM for the influence of aggressive environments (Table 2). The relative mass

Table 2 *Influence of aggressive environments on the mass changes of specimens*

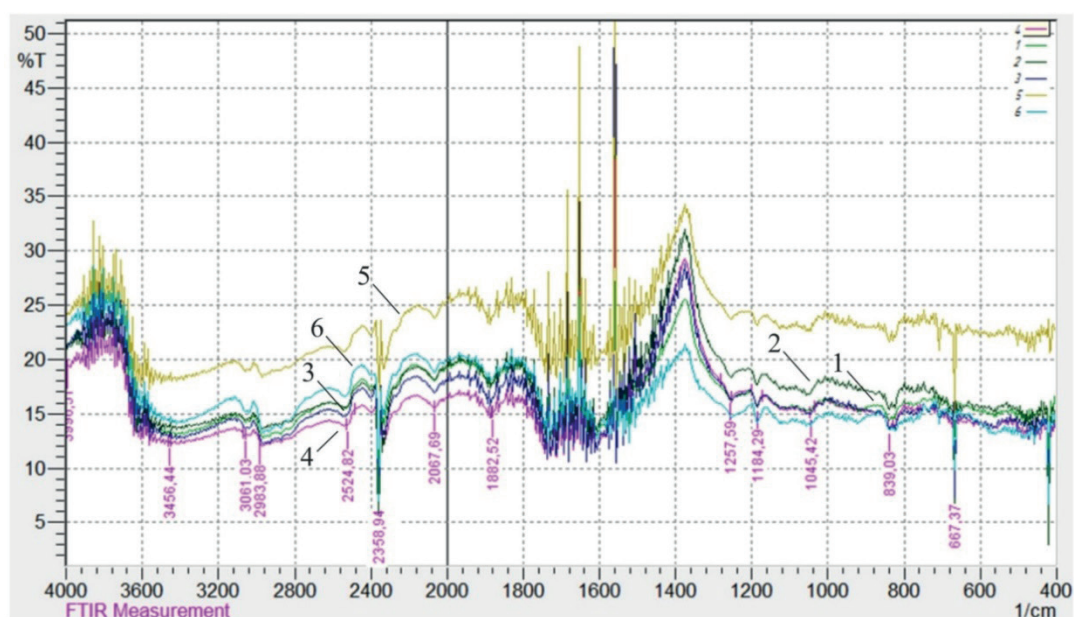| environment	mass changes (%)
air	0.12
oil	0.09
seawater	0.20
river water	0.23
petrol	0.11
NaOH (50%)	0.07

Figure 3 *IR-spectral analysis of CM, which were kept in the aggressive environment:*
1 - air; 2 - oil; 3 - seawater; 4 - river water; 5 - petrol; 6 - NaOH (50%)

change of specimens, which were kept in oil, seawater, river water, petrol, sodium hydroxide, was found.

The specimens, which were kept in oil, are characterized by the low mass change (compared to control the specimen). This is due to the high viscosity of the oil environment, which slows down the process of oil adsorption into the material. At the same time, material, which was kept in petrol, has a comparatively similar relative mass change compared to specimens, which were kept at air. It was believed, that a decrease in the mass change was due to the evaporation of petrol from the surface of the specimen during the drying. Thus, the maximum mass change was observed for specimens that were kept in sea and river water. It should be noted that the increase in the strength of the specimen, which was kept in seawater, at the moment of destruction (by the second method, Figure 1), can be due to increased flexibility of the macromolecular chains, which contributes to the alignment of stress in the polymer and facilitates the orientation of its molecules at stretching [9]. At the same time, seawater has positive affect on the composite properties (the presence of salt solution), because their water absorption decreases (compared to the river water). The chemical interaction of the aggressive alkaline environment with the specimen is in addition weakly expressed, since the reactivity of the material is limited by the slow diffusion of the environment into the material in the solid phase (in the presence of fillers).

The study results of the developed materials' resistance, to influence of the aggressive environment of physical and chemical nature, confirm the presence in the CM of spatial three-dimensional structure. As a result, the polymer loses the ability to dissolve in physically and chemically active environments [9]. The low values of water absorption of all the studied specimens can be explained by the presence of fillers (mica, hexagonal boron nitride), which are characterized by high modulus and low relaxation rate [14, 21-22]. Reducing of the impact strength and energy, spent on destruction of the specimen, which was kept in petrol, can be due to the wash-out of the low molecular weight fraction of the polymer [9].

The comparative IR-spectral analysis of physical and chemical processes on the CM surface, after keeping in aggressive environments, was conducted (Figure 3). The transmittance spectra allowed to determine the following parameters: absorption band intensity (T, %) and the absorption peak area (S, %), which characterize a different number of chemical bonds in polymer and,

Table 3 Characteristic absorption bands and their intensity parameters according to the IR-spectral analysis

band characteristics		IR-spectra of composite materials, which were kept in aggressive environments during 30 days											
group	v (cm^{-1})	No. 1		No. 2		No. 3		No. 4		No. 5		No. 6	
		T (%)	S (%)	T (%)	S (%)	T (%)	S (%)	T (%)	S (%)	T (%)	S (%)	T (%)	S (%)
-NH-, -CH- rocking vibrations, -C-C- stretching vibrations, primary amines: CH$_2$-NH$_2$, CH-NH$_2$, epoxy cycle	839.03	13.7	9.9	13.9	10.0	12.8	7.8	13.7	7.9	21.6	10.1	13.7	10.0
-C-C-, -C-N-, -C-O- stretching vibrations, secondary amines: CH$_2$-NH-CH$_2$, primary amines: CH$_2$-NH$_2$	1184.29	15.7	21.2	16.0	21.0	14.9	20.0	14.7	20.2	23.1	21.2	15.7	21.2
-OH- bending vibrations, -C-N-, -C-O- stretching vibrations; epoxy cycle, primary amines: -NH$_2$	1257.29	16.4	20.5	16.9	20.5	15.6	18.2	15.4	18.5	23.6	20.5	16.4	20.5
stretching vibrations of epoxy group	1882.52	14.9	14.8	16.0	14.8	14.1	14.0	14.1	14.2	23.2	14.2	16.2	14.8
-C≡N- stretching vibrations; alkyne group: -C≡C-H	2067.69	15.4	15.4	15.9	15.4	14.7	14.1	14.4	15.2	23.8	11.1	16.4	15.4
-C≡N- stretching vibrations	2524.82	13.9	7.1	14.1	7.1	11.0	6.0	13.2	6.1	20.7	6.6	14.9	7.1
-CH- stretching vibrations, methyl radical: CH$_3$-C, methylene -CH$_2$-	2983.88	12.4	10.3	13.2	10.3	11.7	10.0	11.4	10.1	18.4	9.7	13.4	10.3
-CH-, -OH, -NH - stretching vibrations, para-benzene	3061.03	12.9	10.2	13.3	10.2	11.0	10.0	12.9	10.2	8.9	9.4	13.8	10.2
-OH, -NH- stretching vibrations	3456.44	12.3	90.1	13.2	90.1	12.3	90.0	12.3	90.1	18.0	85.4	13.7	90.1

consequently, the cross-linking degree of the studied material.

The chemical activity of the surface of the developed material, which was kept in different environments, was conducted by the IR-spectral analysis (Figure 3). Defined adsorption bands correspond to the specific vibration of the molecular units of the epoxy-polyester composite (Table 3). The main difference between the spectra was observed in the region of the absorption bands corresponding to - OH stretching vibrations. These groups are centers that are characterized by increased adsorption and catalytic activity [9]. The activity of the specimen surface, after the action of aggressive environment, is possible to estimate by increasing of the hydroxyl groups, in the near-surface layer. An increase in the absorption band intensity at v = 3456.44 cm^{-1} from T = 12.3-13.7 % to T = 18.0 % appears only for specimen, which was kept in the aggressive environment of petrol. At the same time, decreasing in the absorption peak area (S, %), which is proportional to the number of active groups, was observed. It was considered that the matching of absorption peaks and absence of their displacement, for all the studied specimens, indicates stability of the material surface to chemical interaction with the aggressive environment and, as a result, does not lead to an increase of the sorption of the environment into the material. The comparative analysis allowed to determine the slight difference in intensity of the

absorption band and peak area for all specimens, except specimens kept in petrol.

Based on the conducted studies of the impact strength, mass change and IR-analysis, it was found that developed composite materials can be used in all the studied environments, except petrol, as protective coatings for ship hull superstructures and tanks. This is evidenced by a slight decrease in values of the impact strength of specimens, mass change and the data obtained in analysis of the IR-spectra. In particular, the results of the study suggest the ability of the developed CM to resist the aggressive environment in which the specimens were kept.

4 Conclusions

The impact strength of developed composite materials, which were kept in aggressive environments, was investigated by two methods. It was found that the aggressive environments of oil, seawater, river water, petrol and sodium hydroxide do not significantly affect the ability of polymeric materials to resist shock loads. The impact strength was W = 2.7 - 4.2 kJ/m^2 by the first method (W" = 6.0 - 13.0 kJ/m^2 - by the second method). The energy spent on the specimen destruction varied at E = 0.8 - 1.0 J. The phases of growth of the main crack: the initiation of the crack growth, stable crack propagation and unstable growth of the crack were determined on the "load-time" and "load-deflection" curves. The time of the macrocrack growth initiation, for all the specimens, was τ = 0.07 - 0.16 µs. The maximum load that leads to destruction of a specimen was observed for the control specimen - P$_{max}$ = 2.47 kN. The critical deformation at this load was l = 0.92 mm.

The relative mass change of specimens that were kept in different aggressive environments was found. It was determined that the composite material at the content q = 60 wt% of hexagonal boron nitride and q = 20 wt% of mica is characterized by a decrease in adsorption of environment on the material's surface.

The obtained results were confirmed by the IR-spectroscopy. It was found that the studied composite material is characterized by the same bonds after keeping in different environments. The increase in the intensity of the hydroxyl groups and hence the activity of the material surface, was found for the specimen, which was kept in an environment of petrol.

The results of investigation of the impact strength, mass changes of specimens and IR-spectral analysis, suggest the ability of the developed CM to resist the aggressive environment in which the specimens were kept. Consequently, this confirms the expediency of using the composite material and coating on its basis for the protection of parts and mechanisms of sea and river transport.

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