The study of the impact of 4,4'-methylenbis (2-methoxyaniline) on adhesive properties of the epoxy matrix for protective coatings of transport means

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Abstract

It is shown that one of the main means of regulating the structure and properties of polymer composites is their physical modification. It has been proven that the introduction of plasticizers and fillers into the binder makes it possible to improve the properties of materials.

The effect of the 4,4'-methylenebis (2-methoxyaniline) modifier on the adhesion properties of the epoxy matrix has been investigated. The optimal concentration of the modifier has been established, ensuring maximum indicators of the adhesive strength of the matrix when separated from the steel St. 3 base.

The method of IR-spectroscopy established the chemical structure of the modifier, confirming its activity to interact with the epoxy oligomer in the course of polymerization with a binder. This provides obtaining material not only with improved adhesion strength, but also with negligible residual stresses.

Keywords: *adhesive properties, base, epoxy composite, matrix, modifier, residual stresses, strength.*

Introduction

The development of modern industry sets the conditions for improving the reliability of process equipment, especially the means of transport of the oil and gas industry. In this context, it is advisable to use polymer composite coatings to protect parts of the transport. Recently two – or multi-layer functional coatings are widely used. At the same time, the first layer is important, which performs the function of adhesive. It is shown [1–4] that the adhesive properties of coatings is one of the most important characteristics of the durability of materials during equipment operation. Therefore, the development of protective coatings with enhanced adhesive properties is an important task of protecting equipment from corrosion and wear.

It is known [1–4] that among the existing polymers there are marked materials based on epoxy binder with improved complex properties. An epoxy oligomer is characterized by reactive epoxy and hydroxyl groups that can react chemically with a hardener, resulting in the formation of a network structure of a composite material (CM). To improve the properties of the CM based on epoxy resin, modifiers and fillers of various physical nature and dispersity are introduced into the binder [5–8]. The presence of these ingredients at a critical content improves not only the properties of materials, but also increases their cost-effectiveness by increasing the overhaul period of operation. Promising in this regard is the use of 4,4'-methylenebis (2-methoxyaniline) modifier, which contains components, actively interacting with the epoxy binder. The use of this modifier allows not only to change the supramolecular structure of the epoxy matrix, but also to improve its mechanical properties [3, 4, 8].

The purpose of this work is to establish the effect of 4,4'-methylenebis (2-methoxyaniline) modifier on the adhesion strength of the epoxy matrix on the metal base.

Materials and research methods

Based on the above, as the main component for the binder in the formation of the CM there is chosen ED-20 epoxy diane oligomer (the State Standard GOST 10587–84). The structural formula and model of ED-20 epoxy diane oligomer is shown in Fig. 1.

4,4'-methylenebis (2-methoxyaniline) (MBMA) was used as a modifier. The modifier was introduced into the binder at a content of from 0.10 to 2.00 pts.wt. per 100 pts.wt. of ED-20 epoxy oligomer (hereinafter, pts.wt. are suggested at 100 pts.wt. of ED-20 epoxy oligomer). Molecular weight of 4,4'-methylenebis (2-methoxyaniline) is 258.3. Chemical formula is $C_{15}H_{18}N_2O_2$. The modifier is soluble in benzene, ethanol, acetone, slightly soluble in water. The structural formula of the modifier is shown in Fig. 2.

Polyethylenepolyamine (PEPA) hardener (TU 6-05-241-202–78) has been used to crosslink epoxy composites, which enables materials to harden at room temperatures. It is known that PEPA is a low molecular weight substance, which consists of the following interrelated components $[-CH_2-CH_2-NH-]_n$. The structural formula and model of the fragment of PEPA hardener are shown in Fig. 3. Different stages of

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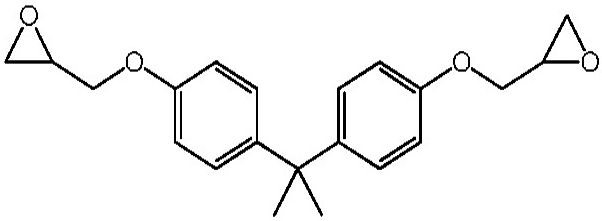


Figure 1 – The structural formula of the fragment of ED-20 epoxy diane oligomer [8]

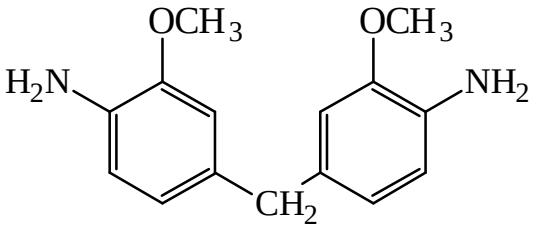


Figure 2 – A general view of the chemical bonds of the 4,4'-methylenebis (2-methoxyaniline) (MBMA) modifier [8]

Table 1 – Characteristics of the components of epoxy binder

Characteristics	ED-20 epoxy oligomer	MBMA modifier	PEPA hardener
Molecular mass	390 – 430	258.30	230 – 250
The content of epoxy groups, %	20.00 – 22.50	–	–
The content of hydroxyl groups, %	1.25	–	–
Average epoxy functionality f_n	2.00	–	–
Nitrogen content, %	–	10.84	19.50 – 22.00
Carbon content, %	–	69.74	–
Hydrogen content, %	–	7.02	–
Oxygen content, %	–	12.39	–
Viscosity η, Pa·s	13 – 20	–	0.90
Density ρ, g/cm^3	1.16	–	1.05

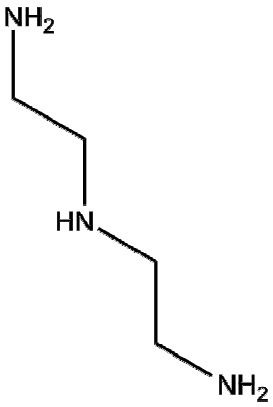


Figure 3 – The structural formula of the fragment of PEPA hardener [8]

crosslinking were modeled and investigated with the introduction of the hardener in the composition for the content of 10 pts.wt. at 100 pts.wt. of ED-20 epoxy oligomer to determine the optimum ratio of components for the corresponding characteristics in the "binder-modifier" system. The characteristics of the epoxy diane oligomer, modifier and hardener are listed in Table 1.

Epoxy composites were formed according to the following technology: heating the resin to a temperature $T = 353 \pm 2$ K and holding at this temperature for a time $\tau = 20 \pm 0.1$ min; hydrodynamic combination of the oligomer and the modifier for a time $\tau = 1 \pm 0.1$ min; ultrasonic treatment (UST) of the composite for $\tau = 1.5 \pm 0.1$ min; cooling the composite to room temperature for a time $\tau = 60 \pm 5$ min; the introduction of the hardener and mixing the composite over time $\tau = 5 \pm 0.1$ min. The CM was hardened according to the following mode: the formation of samples and their exposure for a time $\tau = 12.0 \pm 0.1$ h at a temperature $T = 293 \pm 2$ K, heating at a speed $\upsilon = 3$ K/min to a temperature $T = 393 \pm 2$ K, exposure for a time $\tau = 2.0 \pm 0.05$ h, slow cooling to a temperature $T = 293 \pm 2$ K. In order to stabilize the structural processes in the matrix, the samples were kept for $\tau = 24$ h in the air at temperature $T = 293 \pm 2$ K, followed by experimental tests.

The adhesive strength of the matrix to the metal base was investigated by measuring the breaking stresses with a uniform separation of a pair of glued samples according to the State Standard GOST 14760–69. The study of the adhesion shear strength was carried out according to GOST 14759–69, similarly measuring the force of detachment of adhesion joints of samples by the UM-5 automated tensile testing machine at a loading speed $\upsilon = 10$ m/s. The diameter of the working part of the samples at peeling was $d = 25$ mm. It should be noted that the area of bonding of the samples, which were investigated at peeling and shear, was the same.

The residual stresses in the matrix were determined by the console method [8]. The coating thickness $\delta = 0.3 – 0.8$ mm was formed on a metal base. Base parameters: total length $l = 100$ mm, working length $l_0 = 80$ mm, thickness $\delta = 0.3$ mm.

The deviation of values in studies of indicators of adhesive properties and residual stresses in CM was 4–6 % of the nominal one.

For the study of chemical bonds in the modifier there was used IR-spectral analysis. The IR spectra were recorded on a Fourier Transform Infrared Spectrophotometer IRAffinity-1 (Japan) in the range of wave numbers $\nu = 400 – 2400$ cm^{-1} by the single beam method using reflected light. The sweep of the spectrum by wave numbers $\lambda^{-1} = \nu$ was carried out on a diagram within 225 mm in the range of selected frequencies.

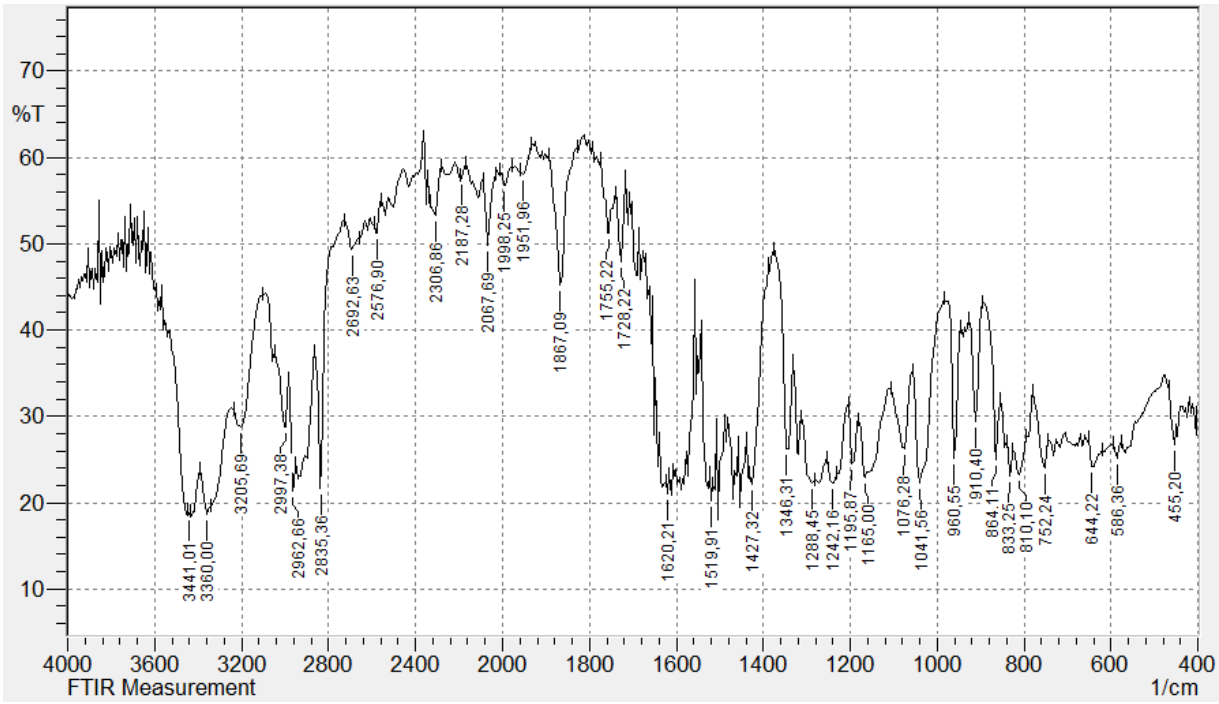


Figure 4 – The modifier spectrum of 4,4'-methylenebis (2-methoxyaniline) in the wave number range ν = 400–4000 cm^{-1}

Wave numbers, transmission intensity, half-width and absorption band area were determined using the IR solution FTIR control software. The error in determining the wave number was ν = ± 0.01 cm^{-1}, and in determining the accuracy of the location of the peak ν = ± 0.125 cm^{-1}. The measurement accuracy was ± 0.2 % with software control of the slot and the integration time t = 10 s. The integration step Δλ = 4 cm^{-1}.

Research results and discussion

Analysis of the works [1–4] allows us to state that during the investigation of the process of crosslinking epoxy composites it is important to directly study the characteristics of the modifier structure, which is introduced into the binder. Therefore, to determine the chemical bonds, the presence of active groups and segments in the structure of the modifier was investigated by the technique of IR spectral analysis. For this, a tablet was formed with an additive without using KBr powder. It should be noted that the transmission intensity was T = 18.0–64.0 %, which indicates the reliability of the experiment (Fig. 4).

The infrared spectrum of the modifier (Fig. 4, Table 2) allowed us to identify the absorption band with the wave number ν = 752.24 cm^{-1}, which has an optical density of D = 24.1 % and a peak area of S = 19.5 %. This absorption band characterizes -NH-, -CH- pendulum oscillations, as well as oscillations of -NH_2 primary amines groups. Absorption bands are also defined, where wave numbers are ν = 810.10–864.11 cm^{-1} with an optical density of D = 23.0–24.9 % and the area of S = 9.3 – 18.6 %, which characterize pendulum oscillations of -NH-, -CH-groups, valent vibrations of -C-C- bonds and primary amines -CH_2-NH_2, -CH-H_2.

Absorption bands at wave numbers ν = 910.40–960.55 cm^{-1} indicate the valent vibrations of -C-C-, -C-N-, -C-O-groups and have an optical density of D = 25.1–29.4 % and a peak area of S = 13.5–16.8 %. If wave numbers are ν = 1041.56–1076.28 cm^{-1}, the absorption bands for the modifier have an optical density D = 22.2–26.2 % and an area S = 26.1–36.3 %. The data in the spectrum refer to valent vibrations of -C-C-, -C-N-, -C-O-groups, tertiary amines $(CH_2)_3N$, and parabenzene.

Valence vibrations of -C-C-, -C-N-, -C-O-groups, secondary amines -CH_2-NH-CH_2- and primary amines CH_2-NH_2 have also been found at ν = 1165.00–1195.87 cm^{-1}. In this case, the band with the wave number ν = 1165.00 cm^{-1} has an optical density D = 22.9 % and area S = 22.9 %, and the band has density D = 24.5 % and the peak area S = 13.0 % at ν = 1195.87 cm^{-1}.

Medium intensity bands have been detected at wave numbers ν = 1242.16–1288.45 cm^{-1}, which have almost the same optical density D = 22.2–22.3 % and area S = 12.0–14.7 %, which indicates the deformation vibrations of OH-groups, valent vibrations of -C-N-, C-O-groups, tertiary -N-R_2 and secondary amines -NH-R. In addition, the presence of the methyl group CH_3-C, the tertiary amines -N-R_2 and -OH-, -CH-groups, have been detected by the presence of an absorption band at the wave number ν = 1346.31 cm^{-1} with the following parameters: D = 22.2 % and S = 19.9 %.

Also, deformation vibrations of -CH-, methylene -CH_2- and methyl CH_3-C-groups have been detected on the modifier spectrum at ν = 1427.32 cm^{-1}. Moreover, this absorption band has an optical density of D = 22.1 % and the area of S = 20.5 %. The absorption

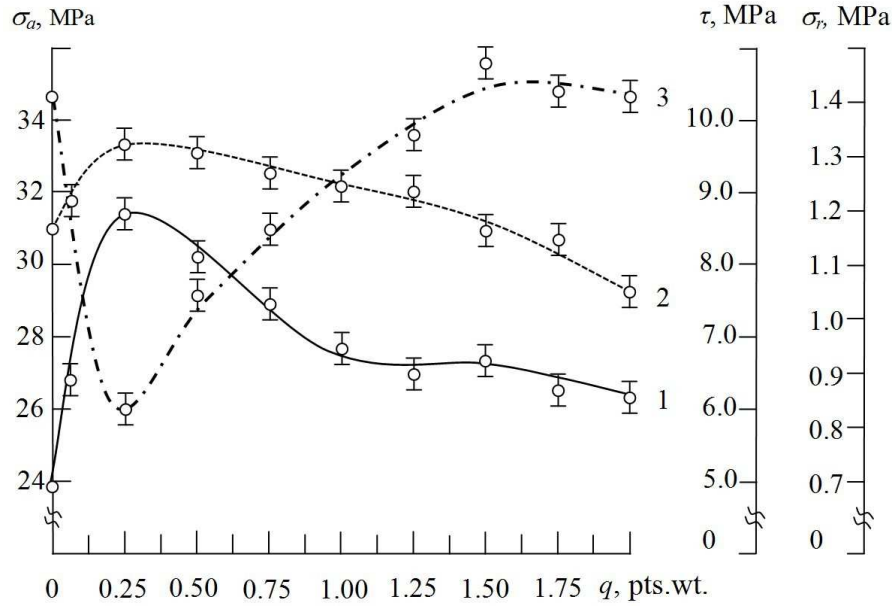
Table 2 – Characteristic absorption bands and parameters of their intensity and area according to the IR spectral analysis of the MBMA modifier

Characteristics of the band		MBMA Modifier	
Group	ν, cm^{-1}	*D*, %	*S*, %
-NH-, -CH- pendulum oscillations, $-NH_2$ primary amines	752.24	24.10	19.50
-NH-, -CH- pendulum oscillations, -C-C- valent oscillations, primary amines: CH_2-NH_2, CH-H_2	810.10	23.20	18.60
	833.25	23.00	9.30
	864.11	24.90	17.40
-C-C-, -C-N-, -C-O- valent oscillations	910.40	29.40	13.50
	960.55	25.10	16.80
-C-C-, -C-N-, -C-O- valent oscillations, tertiary amines: $(CH_2)_3N$, parabenzene	1041.56	22.20	36.30
	1076.28	26.20	26.10
-C-C-, -C-N-, -C-O- valent oscillations, secondary amines: CH_2-NH-CH_2, primary amines: CH_2-NH_2	1165.00	22.90	41.60
	1195.87	24.50	13.00
-OH- deformation oscillations, -C-N-, C-O- valent oscillations, tertiary amines: -N-R_2, secondary amines: -NH-R	1242.16	22.20	14.70
	1288.45	22.30	12.00
-OH-, -CH- deformation oscillations, tertiary amines: -N-R_2, CH_3-C methyl	1346.31	22.20	19.90
-CH- deformation oscillations, $-CH_2$- methylene, CH_3-C methyl	1427.32	22.10	20.50
-NH- deformation oscillations, secondary amines: -HN-R, CH-HN-CH, CH_2-HN-CH_2, parabenzene	1519.21	20.20	5.10
-NH- deformation oscillations, -C=C-, -C=N- valent oscillations, primary amines: CH_2-NH_2-NH_2, parabenzene	1620.21	21.00	24.00
C-O- valent oscillations	1728.22	47.90	6.50
	1755.22	51.20	9.10
	1867.09	45.20	17.00
	1951.96	57.90	6.10
	1998.25	56.70	7.30
	2067.69	49.80	15.20
	2187.28	57.50	5.50
C-N- valent oscillations	2306.86	5320	13.80
	2576.90	51.20	8.50
	2692.73	49.30	20.50
-CH- valent oscillations, $-CH_2$- methylene	2835.36	21.60	5.50
-CH- valent oscillations $-CH_3$-C methyl, $-CH_2$- methylene	2962.66	21.20	20.00
	2997.38	28.70	9.70
-CH-, -OH-, -NH- valent oscillations, parabenzene	3205.69	28.80	9.50
-OH-, -NH- valent oscillations	3360.00	18.70	31.80
	3441.01	18.70	8.40

band for the output modifier at a wave number of ν = 1519.21 cm^{-1} has an optical density of D = 20.2 % and the area of S = 5.1 %, which indicates the deformation vibrations of -NH-groups, secondary amines: -HN-R, CH-HN-CH, CH_2-HN-CH_2 and parabenzene. The average intensity band is detected at the wave number ν = 1620.21 cm^{-1} (D = 21.0 %, S = 24.0 %). This allows us to state the deformation vibrations of -NH-groups, as well as the valent vibrations of -C=C-, -C=N-bonds, primary amines CH_2-NH_2, -NH_2 and parabenzene.

Absorption bands in the range of wave numbers ν = 1728.22–2187.28 cm^{-1} are important, as they indicate the valent oscillations of the C-O-group. In particular, strong intensity bands have been detected at a wave number of ν = 1998.25 cm^{-1}, which has an optical density of D = 56.7 % and the area of S = 7.3 %, and also at ν = 2187.28 cm^{-1}, which has an optical density of D = 57.5 % and the area of S = 5.5 %. This indicates a significant number of these groups in the modifier, which allows us to state the following. This additive in the epoxy matrix can be active before interaction with hydroxyl groups of an epoxy oligomer and the hardener mainly by the C-O-group, since their amount is significant in the modifier.

The formation of strong intensity bands at wave numbers ν = 1206.86–2692.73 cm^{-1} on the IR spectrum of the output modifier indicates the valent vibrations of the C-N-group. Their optical density is not significantly different and amounts to D = 49.3–53.2 %, and the area S = 8.5–20.5 %. This may indicate a significant modifier activity, as an additive, in the chemical interaction with the epoxy oligomer also through these groups, since the intensity of their absorption bands



1 – peeling adhesion strength (σ_a); 2 – shear adhesion strength (τ); 3 – residual stresses (σ_{res}). Base material – the steel St. 3

Figure 5 – Dependence of the adhesion strength and residual stresses in the CM on the content of 4,4'-methylenebis (2-methoxyaniline) modifier

among the entire range of the research spectrum is maximum.

The absorption band at ν = 2835.36 cm^{-1} has an optical density of D = 21.6 % and an area of S = 5.5 %. This band indicates the valent vibrations of the -CH- and methylene -CH_2-groups. It has been established that the absorption bands at wave numbers ν = 2962.66–2997.38 cm^{-1} with an optical density of D = 21.2–28.7 % and an area of S = 9.7–20.0 % correspond to valent vibrations of -CH-, methyl -CH_3-C and methylene -CH_2-groups.

Equally important is the presence of an absorption band at a wave number of ν = 3205.69 cm^{-1}, which has an optical density of D = 28.8 % and an area of S = 9.5 % and corresponds to valent vibrations of the -OH-, -NH-, -CH-groups. It is important to note the presence of absorption bands at wave numbers ν = 3360.00 and ν = 3441.01 cm^{-1}, which are characteristic of valent vibrations of –OH-, -NH-groups. These bands are marked by the same average intensity in the form of optical density D = 18.7 % and the area S = 31.8 % and S = 8.4 %, respectively. It can be argued that there is a small amount of adsorbed water, i.e. -OH-groups in the modifier.

Based on the analysis of the IR spectrum of the MBMA modifier, it can be stated about its activity in the physicochemical interaction with the binder components (ED-20 – PEPA) due to a significant number of groups and segments.

At the next stage, there has been investigated the effect of the content of the MBMA modifier on the adhesive strength at separation (σ_a), displacement (τ) and residual stresses (σ_{res}) in the matrix. It has been established experimentally that the adhesion strength, when the epoxy matrix is detached from the steel St. 3 base, equals σ_a = 24.8 MPa, at displacement – τ = 8.5 MPa, and residual stresses – σ_{res} = 1.4 MPa.

It is shown (Fig. 5) that the introduction of the MBMA modifier into the epoxy binder leads to an improvement in the adhesion properties of materials. In particular, the introduction of an insignificant modifier content (q = 0.10–0.25 pts.wt.) into the epoxy oligomer leads to an increase in the adhesion strength indices when the CM is detached from the steel base from σ_a = 24.8 MPa to σ_a = 26.9–31.3 MPa. With a further increase in the content of the modifier (q = 0.50 pts.wt.), an insignificant decrease in the adhesion strength indices has been observed with the CM separation in σ_a = 28.9 MPa. In the future, the increase in the content of the MBMA modifier from q = 0.75 pts.wt. to q = 2.00 pts.wt. leads to a monotonous deterioration of the adhesive properties of CM to the steel base. In this case, the adhesive strength of the matrix is σ_a = 26.6–28.4 MPa. It can be stated that the maximum increase in adhesion strength in the case of the CM peeling (σ_a = 28.9–31.3 MPa) has been observed for the content of modifier in the matrix in the amount of q = 0.25–0.50 pts.wt. (Fig. 5). In our opinion, with such an additive content, the energy of the adsorption interaction increases, as a result of which physical and chemical bonds are additionally formed. Based on the results of the study of the modifier structure by the method of IR-spectroscopy, it has been believed that with the introduction of an additive at a critical content, the structural network of the polymer is formed with additional chemical bonds as a result of the interaction of -C-N-, -C-C-, -C-O-, -N-H-groups of the modifier with side groups and macromolecule segments of epoxy binder, which is typical with an increase in adhesion rates. The above results of the study suggest the positive

effect of the modifier, especially when it is insignificant, on the course of the matrix formation processes, which, as a result, improves its adhesive properties to the steel base.

A similar tendency has been found on the dependence curve of adhesion strength when peeling the steel St. 3 base (Fig. 5, curve 2). In particular, with the introduction of the MBMA modifier at the content of q = 0.10–0.25 pts.wt. an increase in the adhesion strength from τ = 8.5 MPa (for the initial matrix) to τ = 8.9–9.6 MPa has been observed. Further, with the introduction of the modifier with the content of q = 0.50 pts.wt. an insignificant decrease (within the experimental error) of the adhesion strength at the shear to τ = 9.4 MPa has been observed. It has been proven (Fig. 5), a further increase in the content of the modifier leads to a significant deterioration in the adhesive strength of the CM shear, since the values decrease from τ = 9.4 MPa (at q = 0.50 pts.wt.) to τ = 7.8 MPa (at q = 2.00 pts.wt.). So, the maximum adhesion shear strength (τ = 9.4–9.6 MPa) has been found when the content of the modifier is in the amount of q = 0.25–0.50 pts.wt. It should be noted that the curve of the dependence of the adhesion shear strength on the content of the modifier correlates with the analogous curve of the dependence of the adhesive strength indicators at separation from the concentration of the additive. Given that the maxima in the curves correlate, it is possible to assert the reliability of the experimental data.

To confirm the above provisions, the effect of the modifier content on residual stresses (σ_{res}) in CM was additionally analyzed. It is established that the value of residual stresses in the matrix, treated with ultrasound, is σ_{res} = 1.4 MPa. It is shown (Fig. 5, curve 3) that the introduction of the MBMA modifier in the amount of q = 0.25–0.50 pts.wt. leads to a significant decrease in residual stresses in σ_{res} = 0.8–1.0 MPa. With the introduction of the modifier in the amount of q = 0.75–2.00 pts.wt., residual stresses increase and are σ_{res} = 1.2–1.4 MPa, reaching a maximum at q = 2.00 pts.wt. Analysis of [5–8] suggests that, there is the possibility of peeling or cracking of adhesives at high values of σ_{res}. At the same time, an analysis of the dependences *of* σ_a, τ and σ_{res} on the content of the modifier allows us to establish the optimal content of the additive for the formation of CM with improved properties in the complex. Summarizing the above, it can be stated that the dynamics of residual stresses from the content of the modifier agrees well with the results of experimental studies of peeling adhesive strength and shear strength. However, it should be noted that the maximum values of the complex adhesive properties have been observed for the CM with the content of the modifier in the amount of q = 0.25–0.50 pts.wt. At the same time, such a material is characterized by insignificant indicators of residual stresses σ_{res} = 0.8–1.0 MPa.

Conclusions

On the basis of the conducted studies, the optimal content of the 4,4'-methylenebis (2-methoxyaniline) modifier in the epoxy matrix with improved adhesive properties has been established. It has been proven that to form a matrix with improved adhesion properties to the steel base, it is necessary to introduce a 4,4'-methylenebis modifier (2-methoxyaniline) in the amount of q = 0.25–0.50 pts.wt. into the epoxy oligomer (100 pts.wt.). In this case, the material is formed, which is marked by the following properties: adhesive peeling strength σ_a = 28.9–31.3 MPa, adhesive shear strength τ = 9.4–9.6 MPa, residual stresses σ_{res} = 0.8–1.0 MPa.

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Дослідження впливу 4,4'-метиленбіс (2-метоксианіліну) на адгезійні властивості епоксидної матриці для захисних покриттів засобів транспорту

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Показано, що одним із основних засобів регулювання структури та властивостей полімерних композитів є їх фізична модифікація. Доведено, що введення у зв'язувач пластифікаторів і наповнювачів покращує властивості композитів.

Досліджено вплив модифікатора 4,4'-метиленбіс (2-метоксианіліну) на адгезійні властивості епоксидної матриці, обґрунтовано оптимальну концентрацію для забезпечення максимальних показників адгезійної міцності матриці при відриві від сталевої (марка Ст 3) основи.

Методом ІЧ-спектроскопії встановлено хімічну структуру модифікатора, що підтверджує його активність до взаємодії з епоксидним олігомером у процесі полімеризації зв'язувача. Це забезпечує отримання матеріалу з поліпшеними показниками адгезійної міцності та незначними залишковими напруженнями.

Ключові слова: *адгезійні властивості, епоксидний композит, залишкові напруження, матриця, міцність, модифікатор, основа*.