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Structuring of Modified Epoxy Composite Materials by Infrared Spectroscopy

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Abstract. A comparative evaluation of the structuring processes of the epoxy polymer system with epoxy polymers modified with polyvinyl chloride solution and epoxy composites filled with finely dispersed titanium oxide powder was carried out. Analysis of the infrared (IR) absorption spectra of the studied epoxy polymer and epoxy composite materials showed the presence of deformation and valence vibrations of certain groups of atoms. The oscillations of groups of atoms with double bonds and regions of existence of triple bonds were also revealed. In the region of high frequencies, absorption bands correspond to valence vibrations of groups containing a hydrogen atom. The presence of triple bonds in the epoxy polymer system was determined, indicating unreacted functional groups. This fact corresponds to the low content of the gel fraction of unmodified epoxy polymers after heat treatment and indicates the formation of a system with insufficient chemical bonds. The absorption bands of the epoxy composite material filled with titanium oxide powder are characterized by a lower optical density and a larger peak area compared to the bands of the unmodified epoxy polymer, which indicates the formation of a higher number of crosslinking nodes of the epoxy composite material. The introduction of polyvinyl chloride into the composition of the epoxy polymer system increases the degree of structuring of epoxy polymers. However, a smaller number of formed chemical bonds of the modified epoxy polymer was recorded compared to epoxy composites containing titanium oxide particles. The highest degree of structuring is provided in polyvinyl chloride-modified epoxy composites containing titanium oxide powder due to intensive structuring and formation of double and triple bonds.

Keywords: particle surface modification, finely dispersed filler, titanium oxide powder, polyvinyl chloride solution, optical density, infrared spectrum.

1 Introduction

Increasing the service life of military and transport aviation equipment is strategically essential for developing the economy and ensuring the country's defense capability. The complex impact of atmospheric factors, abrasive particles, and aggressive environments causes intense destruction of the surface of aviation equipment parts, which leads to a loss of structural strength and functionality, requiring the replacement of parts or restoration of damaged surfaces.

The most common way to protect aviation equipment from the complex impact of atmospheric factors is by using polymer-based composite coatings, which have a low density and high resistance to aggressive environments. In particular, compositions on an epoxy binder are technological, which allows modifying the structure of the polymer mesh, introducing dispersed fillers, and forming a coating of a complex profile without using specific and expensive equipment. Epoxy composite coatings provide high adhesive strength, hardness, and chemical resistance, allowing them to protect the outer surface of fuselages and jet engine housings from destruction.

The importance of the work is the study of the peculiarities of structuring the developed wear-resistant epoxy composite coatings intended for aviation equipment, which will increase the service life of the products and reduce the costs of repair and maintenance.

2 Literature Review

According to scientific and technical literature, the most effective way to increase thermal resistance is to create a low-permeable protective layer on the surface of the filler containing refractory compounds resistant to oxidation. Necessary conditions for choosing finish substances are thermodynamic compatibility with all components of the composite and oxidation resistance in the entire working temperature range [1, 2].

Finish substances must provide the following:

- reduction of the level of residual stresses (due to reduction of binder shrinkage);

- complete wetting of the surface of the filler;
- displace water vapor and air from microcracks;
- fill microdefects in the surface layer of the filler.

Finishing treatment in the technology of creating polymer composites is a critical stage since the formed protective layer must relax cracks at the "filler-matrix" interface, transfer mechanical load from the matrix to the filler, maintain chemical and microstructural stability in the entire working temperature range, and be resistant to oxidation. Therefore, the finishing treatment creates an optimal ratio between high and very low matrix adhesion with the filler. High adhesion will contribute to the realization of the physical and mechanical properties characteristic of the matrix with its high crack resistance and fragility. Low adhesion will not lead to the dissipation of fracture energy at the "filler-matrix" interface, which will also not increase mechanical properties [3, 4].

In the last stage, finishing is carried out and used as reinforcing additives. The finishing treatment aims to improve technological properties: facilitate the formation process, protect against mechanical damage during processing, and improve the adhesion of the polymer matrix to the filler when used as a reinforcing component of polymer composite materials. Surface modification of nanofillers can increase the hydrophobicity of fillers, improve interfacial adhesion due to chain entanglement or chemical bonding, and eliminate the loose structure of filler agglomerates [5]. The composition of coupling agents, usually, is an emulsion or suspension consisting of polymer components - emulsifiers, film-forming, antistatic and coupling agents that contribute to the leveling and protection of the filler, improving its wettability.

In order to improve the interphase interaction of composite components, chemical modifications of wood fibers with alkalis and coupling agents are used. When processing fillers with coupling agents, their concentrations are the main parameters. Insufficient content of coupling agents leads to poor adhesion between the fillers and the matrix. On the contrary, an overdose will lead to severe agglomeration of fillers. Therefore, the optimal content of coupling agents is necessary for the best characteristics of composite materials [4].

The surface of metal powders is often covered with silane finishes to increase the adhesive interaction on the interface and reduce moisture adsorption [6]. Treatment of hemp fibers with an alkali solution and then with a solution of KMnO₄ or silane (Si69) increases composites' tensile strength and density based on natural rubber without changing their thermal stability [7]. Polyvinyl alcohol strengthens the interfacial bond between the polymer

matrix and fillers, increasing mechanical properties, thermal stability, and fire resistance [8].

An effective way to improve adhesion between components is to treat fillers with oxidation or ozonation [9]. Ozone treatment is one of the most promising surface treatment methods, as it requires low costs and is an environmentally friendly surface treatment. Ozone treatment can control the amount of oxygen functional groups on the surface of carbon nanotubes according to the concentration of ozone [10].

According to published data, polyimides are widely used as finish substances. After combining with polyimide binders, these finishes ensure the performance of polymer composite materials at elevated temperatures. In the case of using an epoxy binder, the polyimide finish substance contributes to a better hardening of the resin in the boundary layers [11].

The problem of surface modification of fillers is relevant in connection with the wide use of thermosetting resins as binders, particularly epoxy resins [12], the problem of surface modification of fillers is relevant. Information on the surface treatment of fillers for this binder is limited. Also, there is no specific data on the compositions of the decorated compositions and the conditions of their application.

3 Research Methodology

The epoxy-diane resin of the ED-20 (DSTU 2093-92) was selected as the starting material for forming the polymer matrix. Polyethylene polyamine – PEPA (TU 20.1-22944575-002-2017) was used to harden epoxy compositions. Additives-reinforced epoxy polymers are finely dispersed titanium oxide powder (TiO₂), polyvinyl chloride powder, and cyclohexanone.

Infrared (IR) spectra were recorded on an IRAffinity-1S spectrophotometer (Japan) in the frequency range of 400-4000 cm⁻¹ by the single-beam method in reflected light. The spectrum sweep by wave numbers $\lambda^{-1} = v$ was carried out on the diagram within 225 mm in the range of selected frequencies. Wave numbers were determined using the IRsolution computer program. The error in determining the wave number $v = \pm 0.01$ cm⁻¹, determining the accuracy of the peak location ± 0.125 cm⁻¹. The photometric accuracy was ± 0.2 % with software control of the slit and the integration duration t = 10 s. The integration step $\Delta\lambda = 4.0$ cm⁻¹. The material was pre-dried at a temperature $T = (373 \pm 2)$ K for 20 min and mixed in an agate mortar with KBr powder. Then on a hydraulic press with a force F = 20 MPa, samples were formed in the ratio: material under study - 1 mg, KBr - 300 mg.

Quantitative and qualitative analysis (presence of active groups and segments in the structure) of epoxy composite materials was carried out using IR spectroscopy. Most of the IR absorption bands of polymers correspond to specific vibrations of atoms in the polymer chain. IR absorption bands of the same types of vibrations of the atomic group of various substances are located in a specific range of the IR spectrum. The position of absorption maxima on IR spectra characterizes the nature of the substance. The number of absorption bands, their width, shape, and intensity are determined by the structure and chemical composition of the substance. In order to study the relative content of specific functional groups in polymers, the "internal standard" method is used, that is, the ratio of the absorption intensities of two bands, expressed in terms of the optical density (D) and the value of the half-width of the absorption band (b, cm^{-1}) [13].

The content of the gel-sol fraction determined the degree of hardening of materials and coatings. The method is based on the ability of a part of the material (a film) not bound into a polymer mesh to be washed out by an organic solvent in a Soxhlet extractor, which operated in automatic mode.

4 Results

The IR absorption spectra of the developed epoxy polymer and epoxy composite materials were analyzed (Table 1).

Table 1 – Composition and formation technology of epoxy composite materials

Composition No.	Composition and formation technology
1	ED-20 + PEPA
2	$(ED-20 + PEPA) + TiO_2$
3	(ED-20 + PEPA) + (PVH + Cyclohexanone)
	for 1 hour
4	(ED-20 + PEPA) + (PVH + Cyclohexanone)
	for 1 hour + TiO_2
5	(ED-20 + PEPA) +[(PVH +
	+ Cyclohexanone) 1 hour + TiO ₂]
	from surface modification of filler particles
	within 10 min

IR spectra revealed deformation and valence vibrations of certain groups of atoms. In a 400-1500 cm⁻¹ frequency range, there are deformation vibrations of -N-H-, -O-H-, and -C-H- and valence vibrations of -C-C-, -C-O-, and -C-N- groups. Group vibrations of

compounds with double bonds (C=C, C=O, and C=N) are present in a 1500-1800 cm⁻¹ frequency range. These oscillations are not purely valence since adjacent angles and bonds play a large part. The frequency interval of 2000-2300 cm⁻¹ corresponds to the region of existence of triple bonds (C=C and C=N). In a high-frequency region (2700-4000 cm⁻¹), absorption bands correspond to valence vibrations of groups containing a light hydrogen atom (C-H, N-H, and O-H).

For the epoxy polymer sample No. 1, containing an epoxy oligomer and a polyethylene polyamine hardener, the following absorption bands were detected in the area of deformation vibrations (Figure 1):

– optical density D = 0.39 and a peak area S = 13.8 % at frequency v = 561.3 cm⁻¹;

- optical density D = 0.31 and peak area S = 44.8 % at frequency v = 827.5 cm⁻¹;

- optical density D = 0.32 and peak area S = 26.0 % at frequency v = 1035.8 cm⁻¹;

– optical density D = 0.31 and peak area S = 19.7 % at frequency v = 1182.4 cm⁻¹;

- optical density D = 0.30 and peak area S = 16.8 % at frequency v = 1247.9 cm⁻¹;

- optical density D = 0.28 and peak area S = 10.3 % at frequency v = 1454.3 cm⁻¹.

In the range of valence vibrations, there are absorption bands corresponding to double bonds:

- optical density D = 0.27 and peak area S = 10.3 % at frequency v = 1514.1 cm⁻¹;

- optical density D = 0.35 and peak area S = 11.8 % at frequency v = 1606.7 cm⁻¹;

– optical density D = 0.48 and peak area S = 3.5 % at frequency v = 1747.5 cm⁻¹.

They also correspond to triple-bond tongues:

- optical density D = 0.51 and peak area S = 12.0 % at frequency v = 2069.6 cm⁻¹;

- optical density D = 0.51 and peak area S = 24.2 % at frequency v = 2320.4 cm⁻¹).



Figure 1 – IR spectrum of epoxy composite No. 1

In the high-frequency areas, oscillations with optical density D = 0.25 and peak area S = 36.2 % at frequency v = 2924.1 cm⁻¹ were detected. The detected vibrations indicate the presence of -CH- and methylene $-CH_2-$ valence vibrations.

Absorption at frequency $v = 3323.4 \text{ cm}^{-1}$ with optical density D = 0.38 and peak area S = 4.8 % indicates valence vibrations of $-\text{OH}^-$, $-\text{NH}^-$, and $-\text{CH}^-$ groups.

For the epoxy composite material filled with TiO_2 powder (composition No. 2), the following absorption bands were detected (Figure 2):

– optical density D = 0.30 and peak area S = 23.8 % at frequency v = 567.1 cm⁻¹ in the region of low frequencies;

– optical density D = 0.26 and peak area S = 50.3 % at frequency v = 829.4 cm⁻¹;

– optical density D = 0.26 and peak area S = 30.1 % at frequency v = 1037.7 cm⁻¹;

– optical density D = 0.25 and peak area S = 22.9 % at frequency v = 1182.4 cm⁻¹ (the same frequency as the epoxy polymer band);

- optical density D = 0.24 and peak area S = 44.1 % at frequency v = 1246.0 cm⁻¹;

– optical density D = 0.24 and peak area S = 12.7 % at frequency v = 1456.3 cm⁻¹.



Figure 2 – IR spectrum of epoxy composite No. 2

Absorption bands characterize the areas of double bonds shifted toward smaller wave numbers with the following: – optical density D = 0.20 and peak area S = 25.4 %

at frequency $v = 1506.4 \text{ cm}^{-1}$; - optical density D = 0.45 and peak area S = 1.3 %

- optical density D = 0.45 and peak area S = 1.5 %at frequency $v = 1743.7 \text{ cm}^{-1}$.

An absorption band is present at the identical frequency $(v = 1606.7 \text{ cm}^{-1})$ with sample No. 1 but with a lower optical density D = 0.29 and peak area S = 18.7 %.

In the areas of triple bonds, two absorption bands were detected:

- optical density D = 0.46 and peak area S = 45.7 % at frequency v = 2065.8 cm⁻¹;

- optical density D = 0.44 and peak area S = 12.9 % at frequency v = 2362.8 cm⁻¹.

The following shifted absorption bands also characterize the high-frequency areas:

- optical density D = 0.23 and peak area S = 39.4 % at frequency v = 2926.0 cm⁻¹;

- optical density D = 0.33 and peak area S = 10.8 % at frequency v = 3334.9 cm⁻¹.

The absorption bands of identical frequencies $v = 561.3 \text{ cm}^{-1}$ (D = 0.34, S = 15.0 %), $v = 1247.9 \text{ cm}^{-1}$ (D = 0.22, S = 46.6 %), and $v = 1454.3 \text{ cm}^{-1}$ (D = 0.25, S = 11.4 %) with bands of the epoxy polymer sample of composition No. 1 are presented in Figure 3.

There are also bands absorption of the identical frequency $v = 1037.7 \text{ cm}^{-1}$ (D = 0.24, S = 31.5 %) with epoxy composite material composition No. 2, and an absorption band – at the same frequency $v = 1182.4 \text{ cm}^{-1}$ (D = 0.23, S = 23.8 %).

In the areas of double bonds, the absorption bands are shifted towards smaller values of $v = 1504.5 \text{ cm}^{-1}$ (D = 0.18, S = 10.8 %) and $v = 1712.8 \text{ cm}^{-1}$ (D = 0.30, S = 5.7 %). The absorption band is available at the same frequency $v = 1606.7 \text{ cm}^{-1}$ (D = 0.26, S = 14.6 %).

In the region of triple bonds, two absorption bands were detected:

- optical density D = 0.43 and peak area S = 32.3 % at frequency v = 2069.6 cm⁻¹;

- optical density D = 0.41 and peak area S = 11.3 % at frequency v = 2351.2 cm⁻¹.



Figure 3 – IR spectrum of epoxy composite No. 3

The high-frequency areas have the following absorption bands:

- optical density D = 0.24 and peak area S = 38.8 % at frequency v = 2929.9 cm⁻¹;

– optical density D = 0.30 and peak area S = 9.9 % at frequency v = 3321.4 cm⁻¹.

The obtained results indicate that introducing polyvinyl chloride as a modifying additive (epoxy polymer composition No. 3) increases the degree of structuring of epoxy polymers.

For epoxy composite material composition No. 4 in the frequency range 400-1500 cm⁻¹, the following absorption bands were found (Figure 4): $v = 567.1 \text{ cm}^{-1}$ (D = 0.46, S = 3.9 %); $v = 763.8 \text{ cm}^{-1}$ (D = 0.53, S = 8.4 %); $v = 829.4 \text{ cm}^{-1}$ (D = 0.40, S = 21.7 %); $v = 1037.7 \text{ cm}^{-1}$ (D = 0.41, S = 19.3 %); $v = 1182.4 \text{ cm}^{-1}$ (D = 0.39, S = 14.6 %); $v = 1246.0 \text{ cm}^{-1}$ (D = 0.37, S = 29.1 %); $v = 1456.3 \text{ cm}^{-1}$ (D = 0.43, S = 7.5 %).



Figure 4 - IR spectrum of epoxy composite No. 4

In the areas of double bonds, the fluctuations in frequencies $v = 1508.3 \text{ cm}^{-1}$ (D = 0.32, S = 14.6%) and $v = 1705.1 \text{ cm}^{-1}$ (D = 0.51, S = 2.1%) were detected. The absorption band is available at the same frequency $v = 1606.7 \text{ cm}^{-1}$ (D = 0.45, S = 11.5%).

Also, one band with optical density D = 0.65 and peak area S = 11.7 % at frequency v = 2065.8 cm⁻¹ was detected in the areas of triple bonds. Such a band indicates the presence of double bonds of valence vibrations -C=C-, -C=N-, and -C=O- groups.

The following absorption bands characterize the high-frequency areas:

- optical density D = 0.43 and peak area S = 20.3 % at frequency v = 2929.9 cm⁻¹;

- optical density D = 0.50 and peak area S = 2.9 % at frequency v = 3356.1 cm⁻¹.

The absorption bands have higher optical densities and smaller peak areas than materials No. 2-3.

For epoxy composite material No. 5, the following absorption bands shifted to higher values were found in the deformation oscillations (Figure 5): region of $v = 569.0 \text{ cm}^{-1}$ (D = 0.37, S = 5.7 %); $v = 767.7 \text{ cm}^{-1}$ S = 15.5 %); v = 829.4 cm⁻¹ (D = 0.43,(D = 0.32,S = 29.7 %); v = 1037.7 cm⁻¹ (D = 0.33, S = 25.0 %); $v = 1182.4 \text{ cm}^{-1}$ (D = 0.31, S = 19.4 %); $v = 1246.0 \text{ cm}^{-1}$ S = 19.3 %): v = 1456.3 cm⁻¹ (D = 0.31.)(D = 0.34.)S = 9.8 %).



Figure 5 – IR spectrum of epoxy composite No. 5

In the areas of valence oscillations, the following absorption bands at frequencies were found: $v = 1506.4 \text{ cm}^{-1}$ (D = 0.27, S = 21.4 %); $v = 1714.7 \text{ cm}^{-1}$ (D = 0.41, S = 6.3 %). They correspond to the area of the double connections absorption band at the same frequency $v = 1606.7 \text{ cm}^{-1}$ (D = 0.35, S = 16.8 %), comparing the IR spectrum of the epoxy composite material No. 4.

In the areas of triple bonds, absorption bands with optical density D = 0.48 and peak area S = 36.86% were detected at frequencies v = 2065.76 cm⁻¹ and with optical density D = 0.48 and peak area S = 4.15% at frequencies v = 2345.44 cm⁻¹.

The high-frequency areas are characterized by absorption bands with optical density D = 0.36 and peak area S = 21.4 % at frequency v = 2964.6 cm⁻¹, as well as optical density D = 0.38 and peak area S = 7.2 % at frequency v = 3323.4 cm⁻¹.

5 Discussion

The areas of triple bonds of epoxy composite No. 1 are characterized by increased optical density and low peak areas, indicating unreacted functional groups. Absorption bands in these areas are associated with oscillations in which stretching of the corresponding bonds is observed. Double, more stable bonds indicate a complete crosslinking of the epoxy composite material [13, 14].

The content of the gel fraction of unmodified epoxy polymers after heat treatment is the lowest (91.7%), which

indicates the formation of a system with insufficient crosslinking of the polymer matrix.

The absorption bands of epoxy composite No. 2 are identical to those of the epoxy polymer sample composition No. 1, slightly shifted in frequency towards higher values. The absorption bands of the epoxy composite sample No. 2 are characterized by lower optical density and a larger peak area compared to the bands of the epoxy polymer (sample No. 1). This indicates the formation of a higher number of crosslinking nodes of the polymeric material -N-H-, -O-H-, and -C-H-, as well as valence vibrations of -C-C-, -C-O-, and -C-N- groups, which is achieved by introducing a finely dispersed filler in the form of an active type TiO₂ powder.

The shift of the absorption bands on the IR spectrogram of the epoxy composite material composition No. 2 indicates its higher degree of structuring (93.2 %) than the unfilled epoxy polymer composition No. 1 (91.7 %).

The absorption bands of epoxy composite No. 3 have lower optical densities and larger peak areas compared to the epoxy polymer No. 1 and lower peak areas compared to the epoxy composite No. 2. A decrease in optical density indicates the formation of a more significant number of crosslinking bonds of the epoxy polymer material [15].

The obtained results indicate that introducing polyvinyl chloride as a modifying additive (epoxy polymer composition No. 3) increases the degree of structuring of epoxy polymers.

However, after comparing with the epoxy composite material of composition No. 2, a smaller number of formed chemical and physical crosslinking bonds of this epoxy polymer (composition No. 3) is observed.

Simultaneously, the content of the gel fraction is 92.5 %, which indicates a better interaction between macromolecules of epoxy resin and the surface of the filler than the interaction between macromolecules of epoxy resin and polyvinyl chloride.

The identity of most values of the wave numbers of epoxy composite No. 4 absorption bands with the wave numbers of epoxy composite No. 2 is observed. The uniformity of wave values occurs much less when compared with epoxy polymer No. 3.

However, these frequencies have higher optical density values and smaller peak areas. This fact indicates the presence of a significant number of -CH₂-, -CO-, and -CN- valence, -CH-, -NH- pendulum, -OH-, -CHdeformation vibrations, as well as methylene -CH₂- and methyl -CH₃-C- groups in material, which is probably caused by the content of a modifying additive (polyvinyl chloride) and an active filler (fine TiO₂ powder) with a large number of free radicals. This composition of the epoxy composite material ensures the formation of a structure with a gel fraction content of 94.9 %, which is associated with the dominant influence on the processes of TiO₂ powder structuring. This indicates the activation of the course of physicochemical processes due to introducing a modifier and finely dispersed filler into the epoxy polymer system, significantly affecting the polymer matrix crosslinking.

The obtained lower values of optical density and larger peak area on the spectrum of epoxy composite No. 5 indicate a higher degree of structuring (G = 96.8 %) of epoxy composite material No. 5, compared to material No. 4. This indicates a complete passage of reactions in the material of composition No. 5, more complete than those of composition No. 4, with the formation of a more significant number of crosslinking nodes of the matrix, in which double and triple bonds are involved.

6 Conclusions

Epoxy polymers containing an epoxy oligomer and a hardener (polyethylene polyamine) have absorption bands corresponding to double and triple chemical bonds in the range of valence vibrations. The areas of triple bonds are characterized by increased optical density and low peak areas, indicating unreacted functional groups. This affects the formation of an epoxy polymer system with a low content of the gel fraction (91.7 %), which is associated with an insufficient number of chemical bonds between the end groups of the component macromolecules and incomplete structuring of epoxy polymers.

The absorption bands of the epoxy composite material containing TiO_2 powder have a lower optical density and a larger peak area than those of the epoxy polymer system "epoxy oligomer – polyethylene polyamine". This indicates the formation of more chemical bonds due to the introduction of finely dispersed TiO_2 active powder.

The decrease in optical density for polyvinyl chloridemodified epoxy polymers indicates a more complete structure of the system than for unmodified epoxy polymers. However, a smaller number of formed chemical bonds was recorded compared to the epoxy composite containing finely dispersed titanium oxide powder, on the surface of whose particles there is a significant number of active centers. This indicates a better interaction between the epoxy resin's macromolecules and the filler's surface than the interaction between the macromolecules of the epoxy resin and polyvinyl chloride.

The epoxy composite system, consisting of a modified epoxy polymer binder and titanium oxide filler, contains a significant amount of valence ($-CH_2-$, -CO-, and -CN-), pendulum (-CH-, -NH-) and deformation (-OH-, -CH-) vibrations, as well as methylene $-CH_2-$ and methyl $-CH_3-C-$ groups. This ensures an increase in the content of the gel fraction to 94.9 %, which is associated with the activation of the course of physicochemical processes due to the introduction of polyvinyl chloride and an active filler (fine TiO₂ powder) with a large number of free radicals into the epoxy polymer system.

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