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Exploration of Titanium-Based Fine-Particle Additive Influence on Cohesive and Adhesive Strength Enhancement in Epoxy-Polymer Composites

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The study examines how the inclusion of a dispersed powder filler affects the physico-mechanical properties of an ultrasound-modified epoxy matrix. Varying the filler content from 5 % to 60 % by weight in the composite revealed an optimal concentration for enhanced mechanical properties. Introducing the filler at 5 % led to maximum impact strength ($W = 18.47 \text{ kJ/m}^2$) and minimized destructive stresses during bending ($\sigma_B = 51.75 \text{ MPa}$). At 10 % filler concentration, destructive bending stresses increased significantly from $\sigma_B = 48.0 \text{ MPa}$ to $\sigma_B = 74.85 \text{ MPa}$, with impact strength improving from $W = 7.4 \text{ kJ/m}^2$ to $W = 17.42 \dots 18.47 \text{ kJ/m}^2$. Further increasing filler content to 20 - 60 % resulted in a slight decrease in destructive stresses while still surpassing for the filler-free epoxy matrix strength. Optimal modifier content improved adhesive characteristics, achieving a peak adhesive strength ($\sigma_a = 33.4 \text{ MPa}$) at 20 % filler, albeit with residual stresses at 0.34 MPa. Introducing the modifier at 10% increased adhesive strength to $\sigma_a = 28.6 \text{ MPa}$, marking a 1.15-fold improvement over the filler-free epoxy matrix, while reducing residual stresses from $\sigma_{res} = 1.4 \text{ MPa}$ to $\sigma_{res} = 1.0 \text{ MPa}$. Higher filler content (40 - 60 %) led to decreased adhesive strength and increased residual stresses ($\sigma_{res} = 0.62 \dots 0.69 \text{ MPa}$).

Keywords: composite, polymer, powder, filler, structure, strength, adhesion, epoxy resin.

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Introduction

Currently, the industrial and construction materials market offers a diverse selection of synthetic polymers and their composite counterparts, featuring a broad spectrum of properties, including those with special characteristics. Epoxy resin-based composites play a crucial role because of their excellent adhesion to diverse materials, minimal shrinkage during curing, and various other properties. They are widely utilized in different industrial sectors, serving as sealing compounds, immersion resins, coatings, packaging materials, sealants, repair materials, and more [1].

Scientific research in the field of polymer composites based on epoxy compounds and their protective coatings represents is one of the most widespread classes of materials that are actively developing. The characteristics of these composites make them well-suited for the

manufacturing of varnishes, adhesives, impregnants, casting compounds, and various other products. However, in spite of the existence of a large number of composite materials based on epoxy oligomers, their characteristics do not always meet the requirements of modern technology. [2-4].

One of the most widely used and effective methods for producing polymer composites with improved processing and physicomachanical characteristics is to introduce of various fillers with different chemical compositions, morphologies, and dispersions [2-11]. This strategy allows achieving improved characteristics and expanding the range of potential applications for the composites. Epoxies are commonly modified by the inclusion of inorganic-particulate fillers, such as silica [12, 13], alumina [5, 6, 14, 15], mica [7], or talc [2]. Fillers are added to epoxy resins to improve fracture toughness [3, 4] and electrical or heat transfer properties [8, 9], to increase

resin stiffness [10], flame retardance [11], and wear resistance [14], and to reduce the coefficient of thermal expansion (CTE) [16].

Numerous variables, including resin crosslink density, particle type, size, size distribution, and filler loading, can influence the thermal, electrical, mechanical, and fracture properties of the composite. Rubber fillers enhance fracture toughness through mechanisms like cavitation and shear yielding, but they tend to reduce flexural and Young's moduli [17, 18], along with an increase in the coefficient of thermal expansion (CTE). On the other hand, inorganic fillers generally contribute to an increase in modulus and a decrease in CTE, but they typically don't provide as significant an improvement in toughness as their organic counterparts [17].

One of the modern methods for modifying polymer matrix composites (PMCs) is the utilization of carbon nanotubes as fillers. The authors of the study [19] investigated the influence of multi-layered carbon nanotubes in a rubbery epoxy matrix and found that the addition of carbon nanotubes in amounts up to 4 wt.% can lead to a significant increase in strength and Young's modulus.

Polymer composites utilizing ultra- and nanodispersed powders as fillers, characterized by high aspect ratios, also attract considerable interest. Over the past decades, numerous studies have been published, dedicated to exploring the impact of the chemical composition, shape, size, and mass content of powdery fillers on the fundamental structural and physico-mechanical properties of composites.

A notable advancement involves incorporating micron-sized boron carbide (B_4C) particles into a long-fiber composite, where Kevlar 49 serves as a reinforcing component. This results in the formation of a hybrid material suitable for the design of structural and thermal components in engineering systems. The authors demonstrate that the inclusion of B_4C significantly enhances mechanical properties, particularly in the transverse direction concerning the orientation of reinforcing fibers [20].

Furthermore, Boron nitride is considered an attractive candidate because of its high thermal conductivity, high temperature resistance, electrical insulator capabilities, and exceptional resistance to oxidation, in addition to its low weight and moderate cost compared to other ceramic fillers.

In another noteworthy study conducted by the authors [21], the introduction of a hybrid filler comprising a Ti_3C_2 /graphene system into the polymer matrix exhibits a remarkable capability to substantially decrease the wear rate of the composite coating during friction, surpassing a reduction of 80 % compared to a pure epoxy coating. Furthermore, this coating demonstrates impressive anti-corrosive properties for the polymer.

It is important to note that in most publications focusing on the impact of powdered fillers on the properties of polymer composites, only single-phase monopowders of one type of filler were used. However, in the study [17], the effectiveness of using composite powders containing both a plastic metal matrix phase and solid high-modulus compounds as a filler is demonstrated, allowing for an expansion of the range of fillers for

polymer composites.

So, fine-dispersed multicomponent titanomatrix composite material was chosen as the model system because of its effectiveness of using has been demonstrated the previous work [18]. The authors showed that changes in filler content significantly affect the level of adhesive strength and substantially increase the fracture stress indicators of the composite.

Thus, the main goal of this research is to investigate and optimize the filler content for cohesive and adhesive properties of epoxy resin-based composites by introducing a dispersed multicomponent composite powder based on titanium. Additionally, the study explores the influence of dispersed filler content on the structural features of the polycomposite material.

I. Materials and methods

During the creation of polymer composites, the epoxy diene oligomer ED-20 (ISO 18280:2010) was utilized as the foundation, renowned for its exceptional fluidity, which in turn facilitated the development of composites with heightened adhesive, physical and mechanical, and thermophysical attributes [17]. For the polymerization process of the epoxy resin-based materials, we employed a cold-curing polyethylene-polyamine PEPA hardener (TU 6-05-241-202-78).

To produce the epoxy composites, the base resin, hardener, and powdered filler were first mixed, followed by shaping volumetric molds and subsequent curing under specific conditions: the resin was heated to $T = 353 \pm 2$ K and holding at this temperature for 20 minutes; the oligomer and filler were hydrodynamically combining for 1.0 min; ultrasonic treatment (UT) of the composition for 1.5 min. After cooling the composition to room temperature, the hardener was added and the components were thoroughly mixed. Then the composite was cured at $T = 293$ K for 2 hours. In order to stabilize the structural processes within the matrix, samples were kept in air for 24 h at $T = 293$ K for 24 hours before conducting experimental tests.

The proportion of dispersed filler ranged from 5 to 60 parts by weight per 100 parts of epoxy oligomer ED-20.

The following mechanical properties of the composite material were investigated: adhesion strength, residual stresses, fracture stresses, and impact strength.

The fracture stresses in bending were determined using an universal testing machine UM-5 in accordance with ASTM D 790-03 and ISO 527-1, -2, respectively. The specimen parameters: length $l = 120$ mm, width $b = 15$ mm, and height $h = 10$ mm.

The impact strength was studied by the Charpy impact test (40 mm span) after ISO 179-1 on an MK-30 pendulum machine at $T = 298 \pm 2$ K and relative humidity of 50 ± 5 %. The specimen dimensions are $63.5 \times 12.7 \times 12.7$ mm.

In investigating the characteristics of polymer coatings from the obtained composites, steel St 3 with a thickness $\delta = 0.3$ mm was used as a base material.

The strength of the matrix adhesion to the metal base was investigated by measuring the fracture stresses at

uniform tearing of a bonded specimens (the "button test" method) according to ASTM D 897-08. The adhesion shear strength was tested after ASTM D 897-08, similarly measuring the tear strength of the specimen adhesion bonds on a UM-5 tensile machine at the loading speed of 10 m/s. The diameter of the specimen test portion in tear was 25 mm.

The residual stresses in the matrix were determined using the cantilever method. Coatings with a thickness of $\delta = 0.3 \dots 0.8$ mm were applied to a metal base - a 100 mm long plate.

The microstructure of the thermally synthesized composite powder was examined using a scanning electron microscope JEOL Supperprobe 733, while the structure of the obtained polymer materials was investigated using an optical microscope XJL-17AT.

II. Results and discussion

2.1. X-ray analysis and SEM microphotography of the synthesized master alloy.

The X-ray analysis of titanium matrix composites is shown in Fig. 1a. According to the phase diagram, Fe-Si decomposes, forming a eutectic melt at temperatures above 1100°C, leading to interaction between titanium and ferrosilicon, resulting in the formation of a heterogeneous structure. Acceleration of in-situ compound synthesis is achieved through mechanical activation of the initial mixture, reducing particle size and increasing the specific surface area of the powders. This improves their packing and increases contact area, thereby reducing diffusion pathways.

According to the X-ray diffraction pattern (Fig. 1), the alloy is characterized by a significant presence of the silicide phases Ti_5Si_4 and $TiSi$, which dominate the X-ray pattern, likely due to increased Si content resulting in a decreased α -Ti phase fraction.

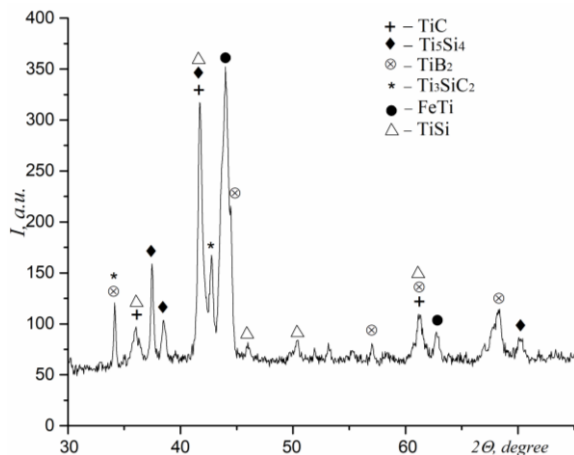


Fig. 1. Fragment of the diffractogram of a TiH_2 -FeSi- B_4C alloy synthesized at 1250°C.

Additionally, peaks of FeTi are observed, possibly formed due to mutual diffusion between Fe and Ti. The presence of Fe significantly promotes Ti- B_4C interaction, resulting in TiB_2 peaks. Diffusion of C and B atoms dissolved in the Fe-Ti melt leads to the formation of the Fe-Ti-C-B melt; upon reaching a certain concentration,

Ti reacts with C and B, indicating the formation of TiC and TiB_2 in the liquid phase.

The X-ray pattern also reveals reflections of titanium silicon carbide Ti_3SiC_2 , with its formation mechanism being complex and dependent on multiple factors. Hypotheses proposed in [22] suggest that the formation of Ti_3SiC_2 occurs due to the depletion of other compounds such as TiC, Ti_5Si_3 , and Si, with the appearance of TiSi indicating certain internal relationships between them. Authors in [23] demonstrated that at synthesis temperatures exceeding 1150°C, the content of titanium carbide decreases, while the silicide phase Ti_5Si_3 disappears, and titanium silicide Ti_5Si_4 and titanium silicon carbide Ti_3SiC_2 appear instead.

The microstructure (Fig. 2) of this master alloy exhibits a porous sponge-like morphology and is characterized by a significantly heterogeneous fine-dispersed structure. The porous structure can be attributed to the formation of micro-pores within the silicide phases, known as the Kirkendall effect, the presence of which results in a reduction in both plasticity and strength values.

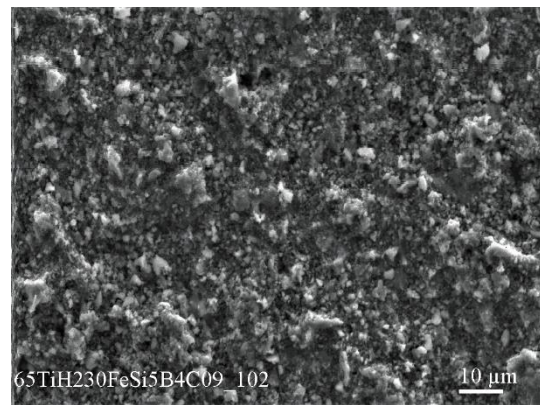


Fig. 2. SEM images of the thermally synthesized multicomponent composite structure.

The brittleness of this alloy is also caused by a significant amount of internal stress, stemming from the large difference in coefficients of thermal expansion, as well as from elastic anisotropy along different crystallographic directions [22]. This property enables the utilization of this alloy as a dispersed filler in complex materials based on epoxy polymers.

2.2 Cohesive properties of epoxy polymer composite.

Investigations into the material's physico-mechanical characteristics were conducted to analyze the influence of the modifier on the cohesive properties of the matrix at various concentrations of the filling component. Before conducting these investigations, the physico-mechanical characteristics of the original epoxy matrix were experimentally determined. To connect the epoxy oligomer with the filler, ultrasonic treatment (UST) was performed, which ensures uniform mixing of components and grinding of particles, which, in turn, leads to uniform mass circulation in the environment of the epoxy binder.

Figure 3 illustrates the dependence of bending strength, σ_B (Fig. 3,a), and impact strength, W (Fig. 3,b), of the composite on the content of the dispersed filler. It is important to note that the ultimate fracture stresses in

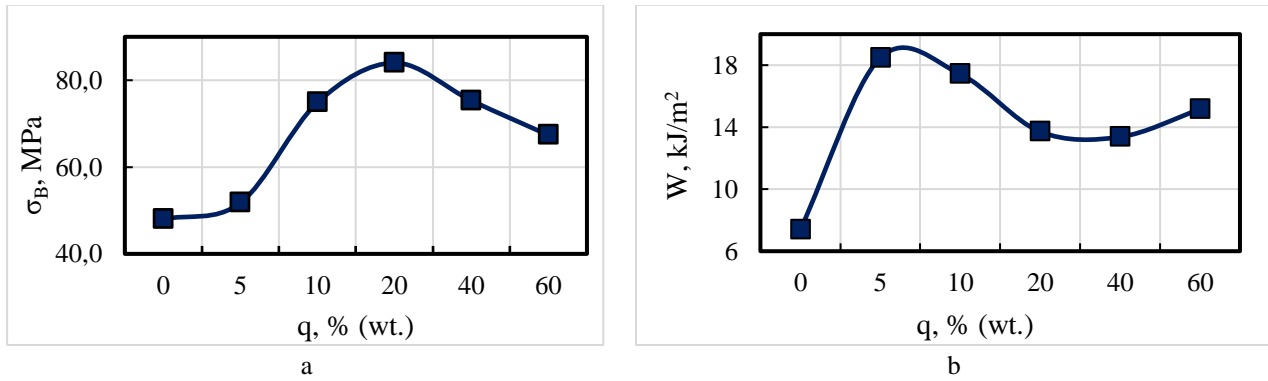


Fig. 3. Bending strength (a) and impact strength (b) of composites vs dispersed filler content.

bending is $\sigma_B = 48$ MPa for the filler-free epoxy matrix. As seen from the Fig. 3,a, the adding of 5 % (wt.) dispersed filler into the epoxy matrix results in the formation of a composite with fracture stresses in bending values that do not significantly differ from those of the original epoxy matrix. A further increase in the dispersed filler content to 10 % (wt.) results in a sharp increase in ultimate bending stress, reaching the value of $\sigma_B = 74.85$ MPa. The maximum bending strength value ($\sigma_B = 84.0$ MPa) is achieved with the introduction of 20 % (wt.) dispersed filler, which promotes an increase in ultimate bending stress by ~ 1.7 times compared to the filler-free epoxy matrix. Further increases in filler content lead to a decrease in the ultimate bending stress within the investigated range of additive concentrations. Nonetheless, the level of these stresses still remains higher than the strength (σ_B) of the filler-free epoxy matrix (Fig. 3,a).

It should be noted that the maxima on the curves of impact strength and bending strength of the composite material significantly differ with respect to modifier concentration. Experimentally, it has been determined (Fig. 3,b) that the filler-free epoxy matrix demonstrates an impact toughness of $W = 7.4$ kJ/m². The addition of the modifier from 5 % to 10 % wt. leads to an increase in the impact strength of the composite. For example, it ranges from 17.42 to 18.47 kJ/m², with the highest value (18.47 kJ/m²) observed for the material containing 5 % filler, which is 2.5 times higher than the initial impact toughness of the filler-free epoxy polymer composite. When introducing from 10 to 60 % (wt.) of the dispersed filler, the impact toughness values begin to decrease, with the lowest value ($W = 13.37$ kJ/m²) observed for the material with a filler content of $q = 40$ % (by weight), yet still exceeding the corresponding characteristic for the original epoxy matrix. Introducing 10 to 60 % (wt.) of the dispersed filler leads to a decrease in impact strength values, with the lowest value ($W = 13.37$ kJ/m²) observed for the material containing $q = 40$ % (wt.) of filler, yet still exceeding the corresponding characteristic of the filler-free epoxy matrix. Therefore, it can be concluded that the ideal range of modifier concentrations for creating matrices with the highest impact strength and bending fracture stress values is $q = 10$ % (wt.).

It is important to note that the decrease in the strength of the polymer composite when exceeding the content of the dispersed component by more than 20 % is associated with various physico-mechanical models presented in a

number of well-known publications by other authors [19-21, 24-26]. One such model, proposed by the authors of [25], focuses on two main factors: the reduction of the cross-sectional area of the matrix carrying the load due to the filling with powder particles, and the level of adhesion at the particle/matrix interface. Another model, discussed in [26], predicts an increase in strength due to a high level of adhesion between the filler and the matrix, but a decrease in strength due to agglomeration exceeding the critical volume fraction of the filler in the material.

When investigating the nature and possible physico-chemical mechanisms leading to the observed regularities, it is also necessary to consider the volume ratio of polymer/filler, the shape and distribution characteristics of its particles, and the interaction at the polymer-filler interface. Developing this concept, authors [3, 24] take into account two main aspects of the filler's influence on the characteristics of the polymer composite - mechanical and structural. The mechanical aspect is determined by the redistribution of stresses during deformation between the matrix and the filler, which contributes to the improvement of mechanical properties. The structural aspect reflects changes in the polymer matrix itself due to interaction with the filler surface, which may affect the rheological and physico-chemical properties of the composite.

Another factor influencing the increase in strength of dispersed-filled composites is the ability of filler particles to undergo elastic deformation. This arises from significantly higher values of the modulus of elasticity in the filler compared to the polymer matrix. Consequently, filler particles undergo minimal deformation under external loading, remaining nearly unchanged due to their substantially higher modulus of elasticity compared to the matrix. This phenomenon induces changes in the stress-strain state in the contact area, leading to alterations in the crack propagation trajectory or even complete crack arrest. In cases where the matrix separates from the filler particles and microcracks form, additional energy is dissipated, promoting energy absorption during failure and enhancing the composite's strength.

Therefore, based on these considerations, it can be concluded that an increase in filler content up to a certain optimal level primarily exhibits a structural influence, driven by modifications in the polymer matrix properties due to interaction with the surfaces of dispersed particles. However, with further increases in filler content, the mechanical effect may become predominant, potentially

resulting in matrix-filler debonding under composite loading.

2.3 Adhesive properties of epoxy polymer composite.

Another important aspect of obtaining composites with high operational characteristics is ensuring significant adhesive contact between the constituents of the heterogeneous system at the phase distribution boundary. In this regard, the analysis of residual stresses after material formation and their relaxation during composite operation is crucial. It is known that residual stresses arise due to interfacial interaction during the formation of physical and chemical bonds between system components, as well as due to volume reduction of composites during shrinkage [17].

Analysis of the obtained research results shows that the adhesive strength (σ_a) of the filler-free epoxy matrix is 24.8 MPa. An increase in the adhesive strength parameter ($\sigma_a = 28.6$ MPa) is achieved with the introduction of a dispersed filler $q = 10$ % (wt.). When the filler content is increased to $q = 20$ % (wt.), a peak is observed on the curve depicting the dependence of adhesive strength on the amount of additive, reaching a maximum value of 33.4 MPa. The improvement of composite properties under such filling is associated with the influence of intermolecular interaction forces of dispersed particles at the "polymer-filler" phase boundary; the ultrasonic treatment modes of the epoxy composition before introducing the hardener are also of significant importance, which determines the formation and interaction of supramolecular structures of the epoxy matrix and filler particles.

Similarly, it is important to note that raising the concentration of the dispersed filler to $q = 40 - 60$ % (wt.) results in a decline in the adhesive properties of the composite material to $\sigma_a = 19.29 - 20.64$ MPa (Fig. 4,a). This decline is evidently attributed to a notable reduction in the specific cross-sectional area of the matrix phase in contact with the substrate material, caused by the infiltration of powder particles.

The results of residual stress analysis, which directly affect their adhesive and cohesive properties, are essential. It should be noted that residual stresses in materials depend on both the nature of the filler and the thickness of

the coatings.

It has been found that the introduction of the modifier (Fig. 4,b) in an amount of $q = 5$ % (wt.) leads to a significant reduction in residual stresses relative to the matrix from $\sigma_{res} = 1.40$ MPa to $\sigma_{res} = 0.29$ MPa. As the amount of additive increases to $q = 10 - 20$ % (wt.), residual stresses slightly increase to $\sigma_{res} = 0.33...0.34$ MPa. It should be noted that in this concentration range of the modifier, composites with maximum adhesive strength properties are formed. This indicates the formation of a strong-bonded structure of the matrix, which improves adhesive properties, despite the slight increase in residual stresses. Further introduction of the modifier, ranging from $q = 40 - 60$ % (wt.), leads to an increase in residual stresses, reaching $\sigma_{res} = 0.62...0.69$ MPa, respectively. The analysis of dependencies between σ_a and σ_{res} on the modifier content allows for determining the optimal additive concentration to create a matrix with enhanced properties across the board. In summary, it can be concluded that the variation in residual stresses with the modifier content correlates closely with the outcomes of experimental investigations into adhesive strength during detachment. At the same time, it should be noted that the maximum values of adhesive properties were observed in composites containing 10 % (wt.) of the modifier.

The microstructure of composites with varying filler contents consists of powder particles ranging from 2 to 5 μm , which are relatively evenly dispersed within the matrix phase. The total surface area occupied by these particles on the section increases with the content of the dispersed component in the composite (see Fig. 5). It is evident that a filler content exceeding 5 - 10 % leads to the formation of powder conglomerates within the composite structure, with both their number and size growing as the filler content increases in the mixture.

For instance, at filler contents of 60 % (wt.), the sizes of such conglomerates reach 20 - 25 μm (Fig. 5,e), significantly contributing to a noticeable decrease in the fundamental mechanical characteristics of the composite.

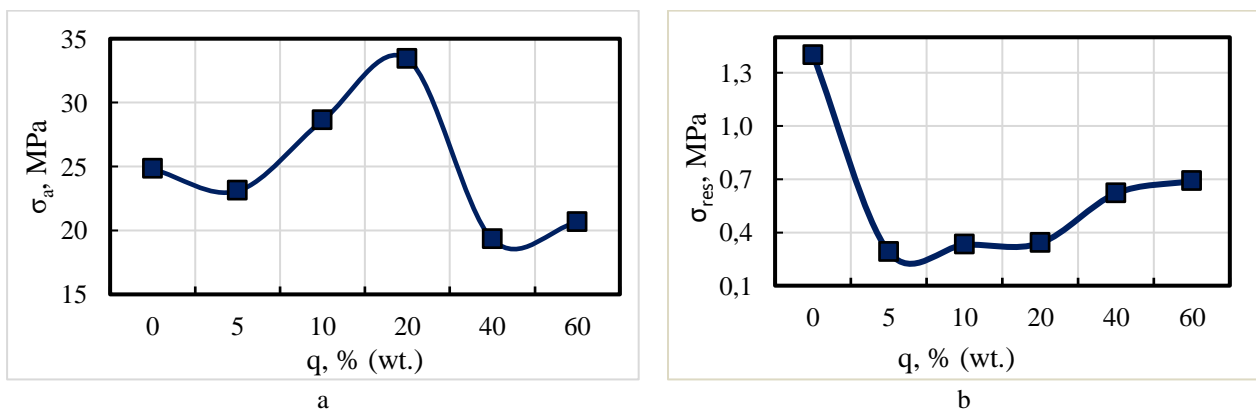


Fig. 4. Adhesion strength (a) and residual stresses (b) in the composite vs dispersed filler content.

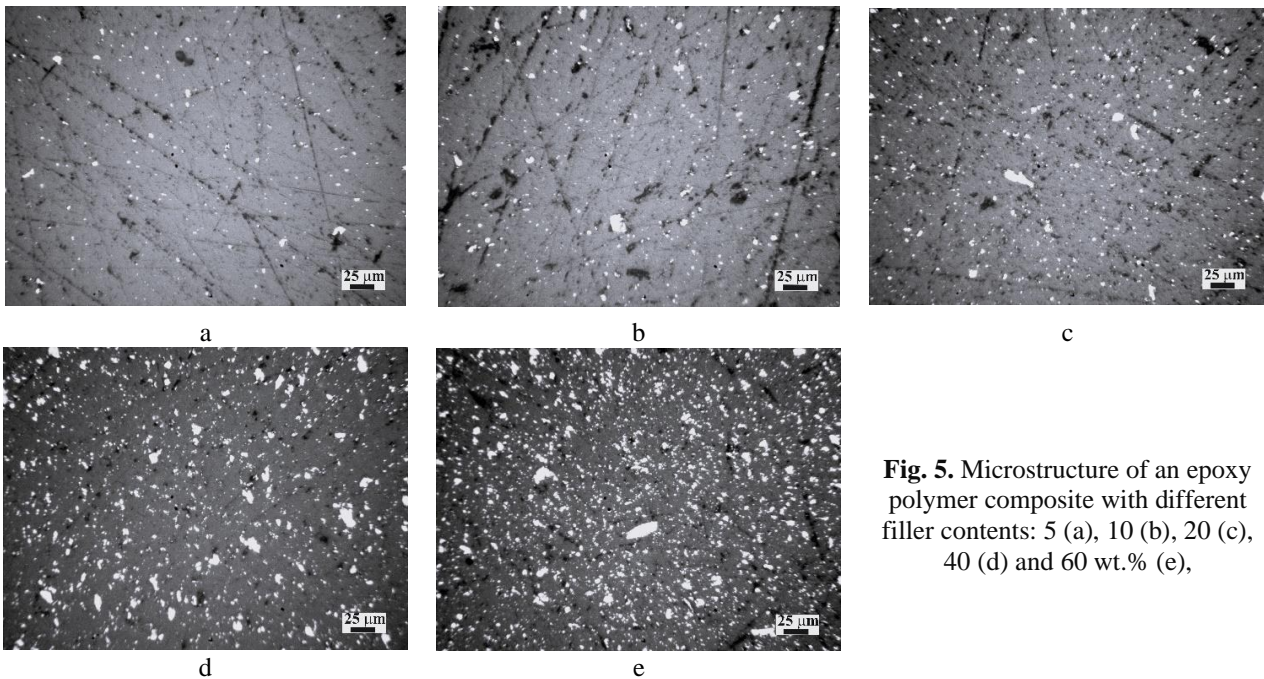


Fig. 5. Microstructure of an epoxy polymer composite with different filler contents: 5 (a), 10 (b), 20 (c), 40 (d) and 60 wt.% (e),

Conclusion

Through thermal synthesis of a powder blend consisting of 65 % TiH₂, 30 % FeSi, and 5 % B₄C (wt.), followed by grinding, a composite powder was produced. This powder is characterized by a complex heterophase system with a significant amount of silicide phases Ti₅Si₄ and TiSi, as well as peaks of FeTi, TiC, and TiB₂, and some quantity of Ti₃SiC₂.

The optimal content of the synthesized powder titanium-based mixture has been determined for forming epoxy composites with improved mechanical properties. It has been demonstrated that filling the composites with dispersed particles at a quantity of $q = 10$ % (wt.) results in increased bending strength of the composite material from $\sigma_B = 48.0$ MPa (for the filler-free epoxy resin) to $\sigma_B = 74.85$ MPa and impact strength from $W = 7.4$ kJ/m² to $W = 17.42 \dots 18.47$ kJ/m². Further increase in the content of the dispersed filler in the composite up to 20 - 60 % (wt.) leads to some reduction in the magnitude of the fracture stresses, however, of the fracture stresses.

Based on the research findings, it's evident that adding a modifier to the epoxy resin at a concentration of

$q = 10$ % (wt.) is crucial for improving the adhesive properties of the matrix. In this case, a material with the following properties is formed: adhesive strength $\sigma_a = 28.6$ MPa and residual stresses $\sigma_{res} = 0.33$ MPa. The highest adhesive strength, $\sigma_a = 33.4$ MPa is achieved with the introduction of 20 % (by mass) of dispersed filler. It has been demonstrated that even a minor amount of dispersion filler ($q = 5$ % (by weight)) leads to a reduction in residual stresses by more than 4 times.

The obtained composite is recommended for use as a matrix in the production of coatings for various functional purposes.

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Дослідження впливу дрібнодисперсних добавок на основі титану на підвищення когезійної та адгезійної міцності епоксидно-полімерних композитів

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Досліджено вплив дисперсного порошкового наповнювача на фізико-механічні властивості модифікованої ультразвуковою обробкою вихідної епоксидної матриці. Змінюючи вміст наповнювача від 5% до 60% (мас.), виявлено оптимальну концентрацію для формування епоксидних композитів з поліпшеними механічними властивостями. Введення наповнювача у кількості 5 % (мас.) приводить до максимуму значення ударної в'язкості ($W = 18,47$ кДж/м²), в той час, коли значення руйнівних напружень при згинанні мають найнижчий рівень $\sigma_{зг} = 51,75$ МПа. Доведено, що наповнення композитів дисперсними частками у кількості $q = 10$ % (мас.) забезпечує підвищення руйнівних напружень при згинанні до $\sigma_{зг} = 74,85$ МПа та ударної в'язкості до $W = 17,42 \dots 18,47$ кДж/м². Подальше збільшення вмісту наповнювача в складі композиту до 20 – 60 % призводить до деякого зменшення величини руйнівних напружень, рівень яких, однак, все ще помітно перевищує рівень міцності вихідної матричної фази. Найбільше значення адгезійної міцності $\sigma_a = 33,4$ МПа досягається при введенні 20 % (мас.) дисперсного наповнювача, але при даному наповненні залишкові напруження становлять 0,34 МПа. При введенні модифікатора у кількості $q = 10$ % (мас.) адгезійна міцність при відриві – $\sigma_a = 28,6$ МПа, що вище у 1,15 рази за значення вихідної епоксидної смоли. Водночас слід зазначити, що введення модифікатора у кількості $q = 10$ мас.ч. зумовлює зниження залишкових напружень від $\sigma_{res} = 1,4$ МПа (для вихідної епоксидної матриці) до $\sigma_{res} = 1,0$ МПа. Збільшення вмісту дисперсної складової до $q = 40-60$ % (мас.) призводить до помітного зменшення рівня адгезійної міцності, а також до зростання залишкових напружень, які становлять $\sigma_{res} = 0,62 \dots 0,69$ МПа відповідно.

Ключові слова: композит, полімер, порошок, наповнювач, структура, міцність, адгезія, епоксидна смола.