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PHYSICO-MECHANICAL PROPERTIES OF EPOXY COMPOSITES FILLED WITH METALLIZED POLYAMIDE GRANULES

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The physical and mechanical properties of epoxy composites filled with copper-plated polyamide granules were investigated. Physico-mechanical properties were evaluated based on the results of tensile and impact toughness studies. It is shown that the obtained composites have high strength properties, which are preserved at the level of the unfilled matrix. It was established that the presence of polyamide granules of a copper shell on the surface has little effect on the change in the physical and mechanical properties of epoxy composites. An attempt was made to explain the obtained results using the values of the strength of the adhesive layer formed between the epoxy matrix and the surface of the filler, which is different in nature.

Key words: composite; epoxy; polyamide; metallization; copper.

Introduction

Epoxy resin is a widely used polymer matrix during the creation of composite materials, which is caused by high adhesion, chemical resistance and relatively low cost. Composites based on epoxy resin are used in the automotive, aerospace, construction and many other industries [1]. As a polymer matrix, epoxy resin can be reinforced with synthetic and natural fillers in various forms (powders, fibers, fabrics, etc.) [2–5]. This possibility of using various fillers is an effective approach for obtaining composites with high operational and technological properties.

Metal fillers are traditionally used to obtain composite materials based on an epoxy matrix. Alloys of iron, aluminum, copper in the form of powder or fibers guarantee the production of composites with increased dimensional accuracy, wear resistance, strength and better tribotechnical properties [6, 7]. Ceramic fillers also help to improve the tribotechnical properties of epoxy composites [8–10]. At the same time, it is possible to achieve a noticeable positive effect with a low concentration of the filler [11]. The filler particles in the epoxy matrix act as reinforcing agents that form a strong interphase bond and effectively transfer loads and prevent crack propagation.

Copper as a filler for various types of polymer matrices is used to improve electrical and thermal conductivity. By improving electrical conductivity and the ability to dissipate heat, copper makes polymer composites suitable for use in electronics. At the same time, copper also increases the mechanical strength and stiffness of polymer composites [12].

In recent years, researchers have widely used various combined fillers, which differ significantly both in nature and in possible influence on the properties of composites [13–15]. In this case, it is possible to obtain promising materials with a complex of new properties. The use of additional carbon nanotubes in the amount of 0.5 % in the epoxy-Kevlar composite allows to improve its tensile strength by 25 %. In addition, the use of carbon nanotubes ensures an increase in the electrical conductivity of the composite, which makes it possible to monitor the growth of cracks by registering changes in the resistance of the material [16]. In the case of a combination of glass fiber and polytetrafluoroethylene particles in an epoxy matrix, a composite characterized by a low wear rate can be obtained, which is especially important for friction nodes [17]. Also, an epoxy composite containing carbon fabric and carbon nanotubes is recommended for use in friction nodes [18]. A composite material characterized by high high-temperature mechanical properties was obtained as a result of the combination of carbon fiber and highly dispersed aramid nanofibers in an epoxy matrix [19]. The possibility of creating a composite material with an epoxy matrix and a filler consisting of sisal fibers and peanut shell particles is considered [20].

An interesting approach to creating combined fillers is the use of a binary system of spherical Al_2O_3 particles that differ in size (30 and 5 μm). At the same time, the epoxy composite containing 80 % of 30 μm particles and 20 % of 5 μm particles is close to the theoretical maximum packaging volume and is characterized by significantly reduced viscosity and increased thermal conductivity [21]. It is also reported on the use of spherical and fibrous boron nitride for filling epoxy resin [22].

Also, the combined fillers include nickel-coated copper particles obtained in [23], which were used to create composites based on an epoxy matrix. This approach made it possible to increase the tensile strength of epoxy composites with the addition of nickel-coated copper particles by 90 % compared to composites that contained copper particles that were not coated with nickel.

As mentioned above, epoxy composites are widely used in the aerospace industry. However, the use of composite materials in such a critical field, in addition to high mechanical properties, thermal and electrical conductivity, also requires the maximum possible weight reduction of products made of polymer composites. The development of new fillers that will allow to reduce the density of the composite material while preserving other properties is a promising and necessary task.

As a filler for the epoxy matrix, in this work, it is proposed to use copper-plated polyamide granules obtained according to the developed method [24, 25]. The introduction of copper-plated polyamide granules into the epoxy matrix will make it possible to obtain a composite material characterized by a low density, and the presence of a copper layer will provide a number of specific properties possessed by metal-filled composites.

The purpose of the work is to investigate the physical-mechanical properties of the developed epoxy composites, which are filled with metallised polyamide granules.

Materials and research methods

Copper-plated polyamide granules were obtained according to the scheme shown in Fig. 1.

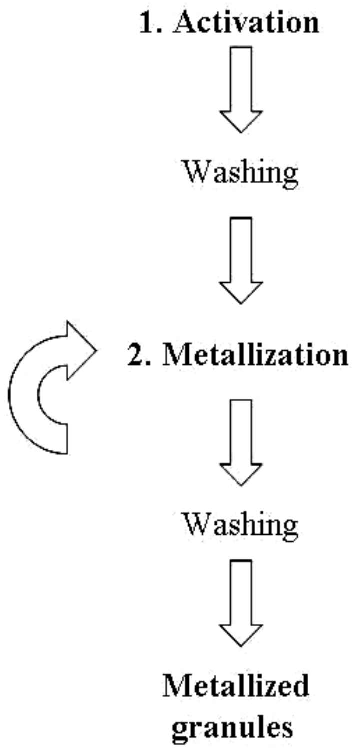


Fig. 1. Scheme of production of copper-plated polyamide granules

Polyamide granules were activated in a ball mill. Polyamide granules, powdered zinc and grinding bodies were loaded into the mill. The rotation speed of the mill is 125 rpm; loaded mass: polyamide – 200 g, zinc – 20 g, grinding bodies – 1.5 kg; processing time in a ball mill is 1 hour. The activation of the polymer surface is necessary for the initiation of the reduction reaction of copper ions in the chemical reduction solution and occurs as a result of fixing zinc particles on it. The washing stage after activation is necessary to remove unattached zinc particles from the polymer surface and to increase the efficiency of metallization [26].

Metallization of activated polyamide granules was carried out in non-stabilized chemical reduction solutions of the following composition (mmol/l): $CuSO_4 \cdot 5H_2O$ – 60, EDTA-Na_2 ($C_{10}H_{14}N_2Na_2O_8 \cdot 2H_2O$) – 67, NaOH – 375, formalin – 365. Granule loading density – 10 g/100 ml of solution. During metallization, the solution was stirred. The time of completion of metallization was determined by the disappearance of the color of the solution. In order to obtain different contents of copper on polyamide granules, the method of repeated metallization was used. For which the already metallized granules were placed in new portions of the chemical reduction solution. Metallized polyamide granules were used as a filler in the preparation of epoxy composites. The content of copper on metallized polyamide granules was: 5 wt. % for composite 1; 7.5 wt. % for composite 2; 10 wt. % for composite 3.

Tecomid NB40 NL E polyamide granules were used for research. Epoxy resin (ED-20) and hardener (polyethylene polyamine) did not contain solvents. Epoxy resin and hardener were mixed in a mass ratio of 10 parts of resin to 1 part of hardener.

The epoxy matrix was filled with original and metallized polyamide granules with the aim of obtaining the maximum filling with granules of the form (which is about 50–60 % by weight). After mixing all components, the liquid composition was vacuumed to remove air bubbles.

Samples for research were obtained by pouring a liquid composition into molds obtained by 3D printing. For printing 3D forms, polylactide was used, the wall thickness of the form is 0.5 mm (Fig. 2, *a*).

a

b

Fig. 2. An example of a form obtained by 3D printing for forming a sample intended for tensile strength studies (a) and a sample prepared for studies (b)

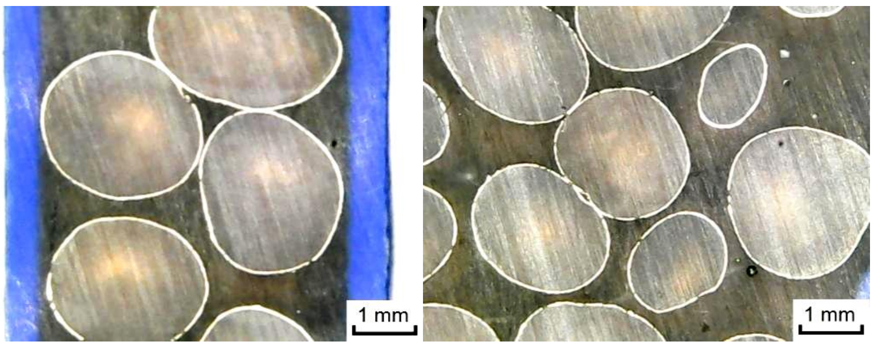


Fig. 3. Distribution of copper-plated granules in the epoxy matrix (the photos were obtained after grinding the samples and the copper shell is visible on them)

The form for pouring samples of the tensile strength study was obtained in accordance with the requirements of ISO 527-2:2012 (type 1BA). For impact strength testing according to ISO 179. After approval, the samples were ground on both sides of Fig. 2, *b* to obtain the dimensions defined by the standards. The distribution of copper polyamide granules in the epoxy matrix is shown in Fig. 3.

Adhesive strength was determined by the pull-off method. At the same time, the magnitude of the destructive tensile force of the butt-glued sample was determined by forces directed perpendicular to the plane of gluing. Cylinders with a surface area of 100 mm^2 were used for research. Options for studying the adhesive strength of various materials are shown in Fig. 4. The polyamide copper plate (item 5 in Fig. 4) was obtained similarly to the described method of granules metallization.

Diffraction curves were recorded under static conditions using an AERIS Research diffractometer (Malvern PANalytical) in symmetric reflection mode.

The WAXSFIT program [27] was used to determine the degree of crystallinity of polyamide and structural parameters of crystals.

The density was measured by the pycnometric method. All studies were performed at least 72 hours after receiving the samples.

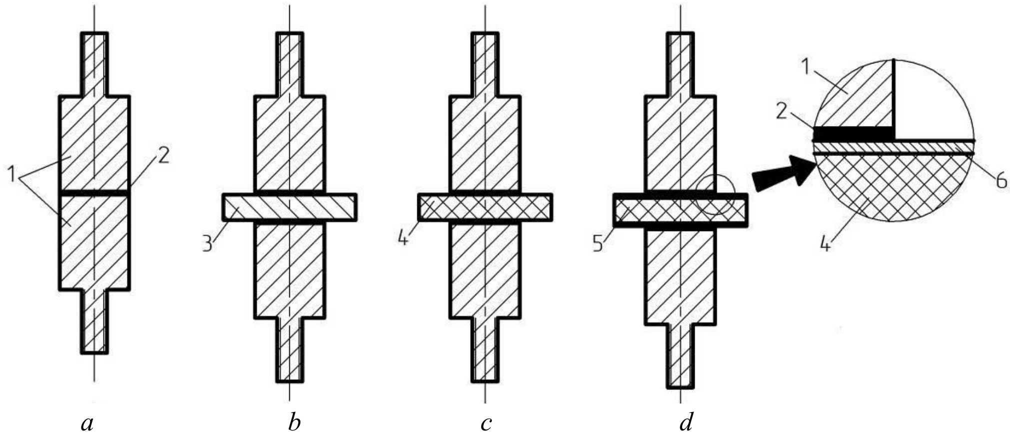


Fig. 4. Variants of gluing materials for establishing adhesive strength:
a – study of the strength of the adhesive seam; b – study of the strength of the adhesion of the epoxy matrix to the copper plate; c – study of the strength of the adhesion of the epoxy matrix to polyamide; d – study of the strength of the adhesion of the epoxy matrix to metallized polyamide:
1 – cylinder; 2 – adhesive seam; 3 – copper plate; 4 – polyamide plate; 5 – metallized polyamide plate; 6 – copper layer formed on the polyamide plate as a result of metallization in a chemical reduction solution

Results and discussion

The formation of a continuous copper shell on the surface of polyamide granules can be established visually (Fig. 5). All granules that underwent copper plating in chemical reduction solutions are covered with a continuous copper shell.

In addition, the formation of polyamide granules on the surface of the copper shell can be confirmed by the results of X-ray structural studies of the original and copper-plated raw materials (Fig. 6).

It is possible to observe the appearance of peaks corresponding to the crystal lattice of copper in the diffractogram of copper-coated granules, which are absent for the original polyamide granules.

It is necessary to note one feature that was discovered during X-ray structural studies of activated polyamide granules. Decrease in intensity of peaks from the crystalline phase of polyamide (Fig. 7).

Production of copper-plated polyamide granules, before metallization, includes an activation stage as a result of granule processing in a ball mill. During activation, zinc particles are fixed on the surface of the granules as a result of the impact of the grinding bodies placed in the ball mill.

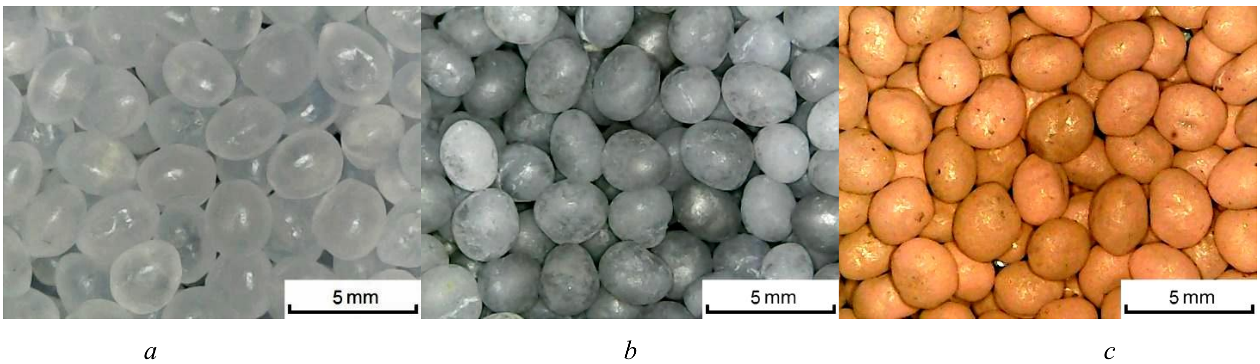


Fig. 5. Photographs of initial (a), activated (b) and copper-plated polyamide granules (c)

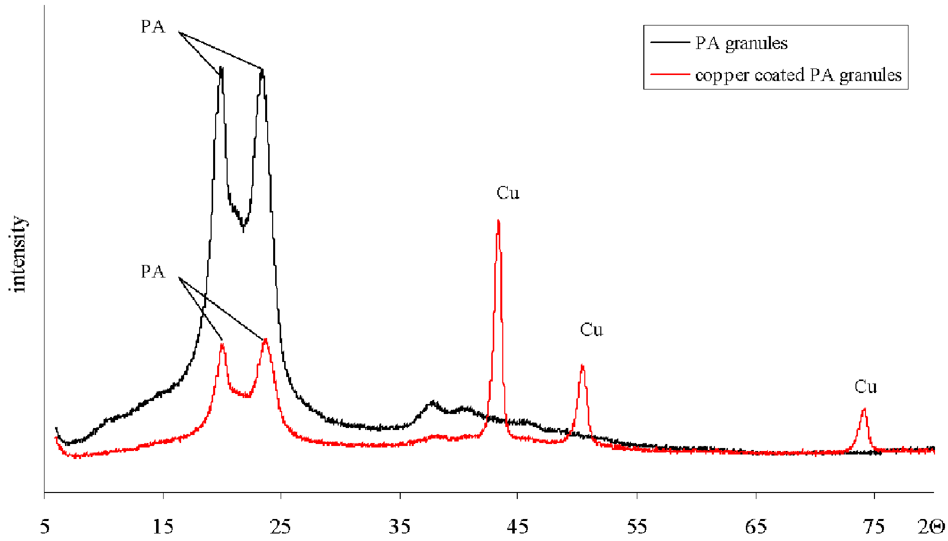


Fig. 6. Diffractograms of initial and copper-coated polyamide granules

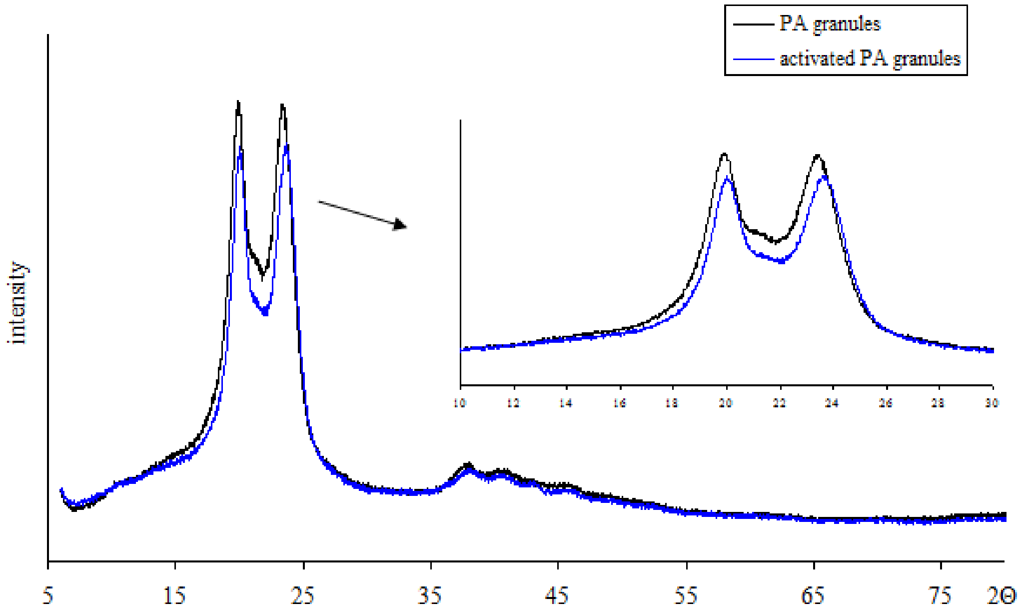


Fig. 7. Diffractograms of initial and activated polyamide granules

The result of such an impact is the accumulation of internal stresses in the granules, which are accompanied by the destruction of crystallites. The broadening of the peaks on the diffractograms of metallized polyamide can be caused by a decrease in the size of the crystallites and the accumulation of lattice deformations [28]. It can be argued that the result of the metallization of the granules (activation stage) is a certain amorphization of the polyamide, which is indicated by a decrease in the intensity and broadening of the peaks, as well as the calculated crystallite sizes (Table 1). At the same time, the shift in the position of the peaks is insignificant, which indicates the absence of a change in the structure of the polyamide crystal lattice.

As shown by the results of the study of the tensile strength of the obtained composites (Fig. 8), the highest strength is characterized by the epoxy matrix. In this case, the form obtained by 3D printing was filled with epoxy resin without the introduction of a filler. The composition with original polyamide granules is characterized by the lowest strength. The use of metallized granules as a filler leads to an increase in the tensile strength of composites, although it does not reach the results obtained for the epoxy matrix. At the same time, the strength of composites with metallized granules increases as the metal content on the polyamide granules increases. Such an increase in strength can obviously be explained by an increase in the roughness of the polymer (and then the copper) surface, which increases with an increase in repeated metallizations.

Table 1

Results of calculations of parameters of crystalline peaks and amorphous phase of polyamide granules

Sample	Degree of crystallinity	Position, 2·θ	Height, units	Width, 2·θ	Size L1, nm	Size L2, nm	Distance *d*, nm
Initial PA granules	0.470	10.7	5.3	6.0	14.8	10.8	8.3
		13.1	1.4	2.1	43.3	30.6	6.8
		15.0	4.8	3.3	27.2	25.5	5.9
		19.9	70.2	1.2	73.4	47.8	4.5
		21.2	35.1	5.3	17.0	16.0	4.2
		23.5	67.6	1.6	56.2	46.1	3.8
		23.0	22.9	28.0	–	–	–
Activated PA granules	0.420	10.4	4.3	6.0	14.8	10.8	8.5
		13.1	1.0	1.5	59.4	55.8	6.8
		15.0	2.1	2.9	30.4	28.6	5.9
		20.0	58.3	1.6	57.4	37.7	4.4
		21.8	9.6	5.7	15.9	14.9	4.1
		23.5	62.7	2.1	43.4	34.9	3.8
		21.8	24.9	28.0	–	–	–

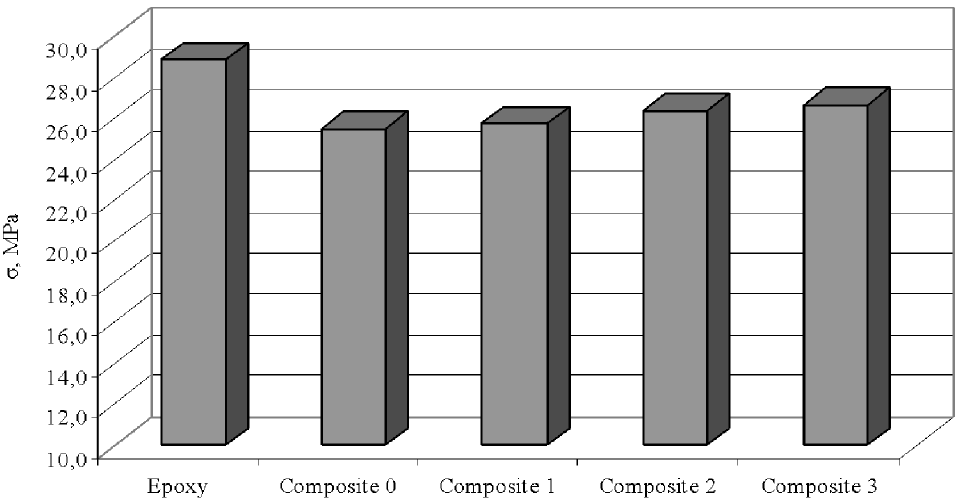


Fig. 8. Tensile strength of composites with an epoxy matrix

It should also be noted that the maximum reduction in the strength of the composite filled with the original granules is insignificant and amounts to about 10–12 %. For composite 3, which is filled with granules with the highest copper content, the reduction in strength is 7 %.

We see a different situation when studying the impact toughness of the obtained composites (Fig. 9). In this case, the presence of filler increases the value of impact viscosity. Moreover, the impact toughness increases both for the original and metallized polyamide granules.

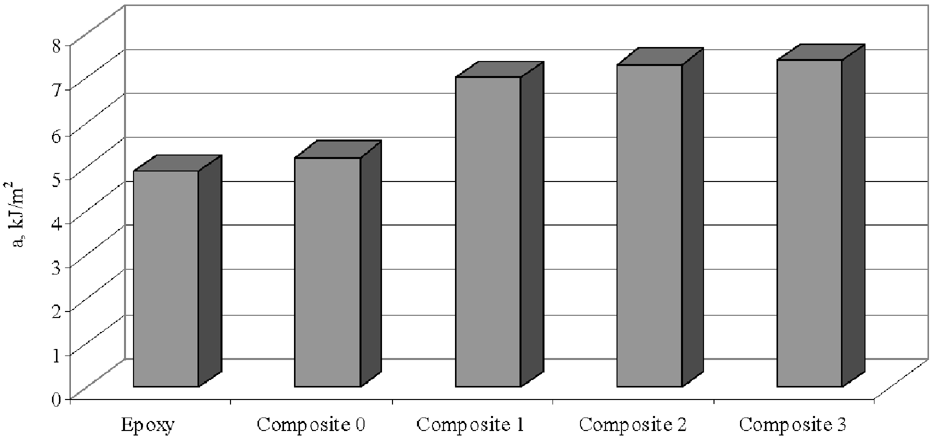
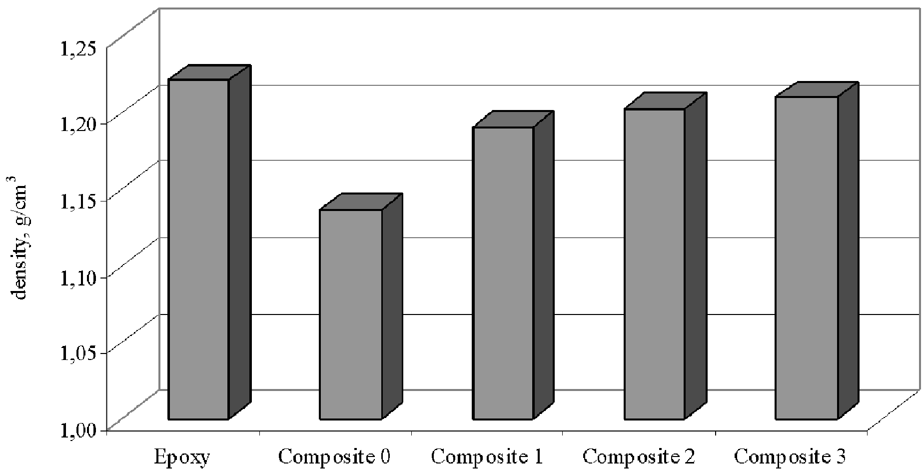
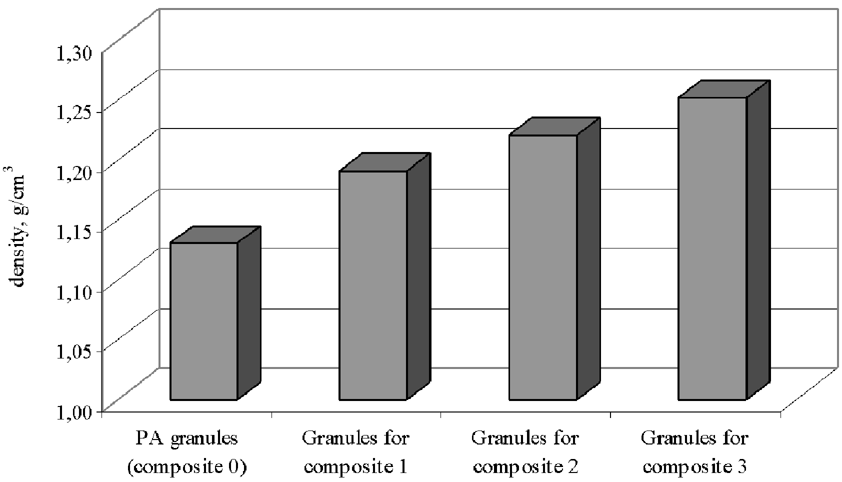


Fig. 9. Impact toughness of composites with an epoxy matrix



a



b

Fig. 10. Density of composites with an epoxy matrix (a)
and polyamide granules used as fillers (b)

Table 2

Adhesive strength

Adhesion	The option according to Fig. 4	Strength, MPa
glue seam	a	20.1
epoxy matrix to the copper plate	b	10.1
epoxy matrix to polyamide	c	5.2
epoxy matrix to metallized polyamide	d	3.5

In the case of impact toughness, a certain increase in its value can be explained by the reinforcing effect of polyamide granules, which, thanks to high impact toughness and a strong interfacial bond with the epoxy matrix, are able to effectively perceive and dissipate short impact loads. At the same time, the presence of copper shell granules on the surface allows more effective perception and dispersion of impact loads as a result of their redistribution between different layers of the material (epoxy-copper-polyamide). Such an explanation seems logical after analyzing the results of the adhesion measurement (Table 2), in which the value of the adhesion strength of the epoxy matrix to the metallized polyamide surface is the lowest. However, given the large number of metallized granules and their significant surface area, which is covered with copper and interacts with the epoxy matrix, it is possible to distribute and absorb some additional energy.

Also, the results of Table 2 can be used to explain the decrease in tensile strength of filled epoxy composites. In this case, tensile tests take place under conditions of low load application speeds (5 mm/min), which contributes to the formation and propagation of cracks in the interphase regions.

The study of the density of the obtained composites shows that, compared to the epoxy matrix, the filled composites are characterized by a lower density (Fig. 10, *a*), which, taking into account the presence of a copper shell on the polyamide granules and the increase in the density of the granules (Fig. 10, *b*), is a promising option.

Production of epoxy composites characterized by low density and having metal fillers in their structure is interesting from the point of view of production of products with low mass. In addition, due to the presence of a metal filler, they will have a complex of interesting properties that can be used in a number of new industries and applications.

Conclusions

Thus, based on the obtained results, it was established that the use of copper-plated polyamide granules allows to obtain epoxy composites with high physical and mechanical properties and low density. The presence of a copper shell on the polyamide granules increases the impact toughness of the composite and practically does not affect the tensile strength. Obtaining filled composites with a density that practically corresponds to the density of the epoxy matrix allows them to be offered for the manufacture of parts with a low mass, which is interesting for the aviation industry. The presence of copper in composites is also promising for creating effective friction nodes on their basis.

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кафедра теплоенергетики, теплових та атомних електричних станцій

ФІЗИКО-МЕХАНІЧНІ ВЛАСТИВОСТІ ЕПОКСИДНИХ КОМПОЗИТІВ, НАПОВНЕНИХ МЕТАЛІЗОВАНИМИ ГРАНУЛАМИ ПОЛІАМІДУ

Досліджено фізико-механічні властивості епоксидних композитів, наповнених мідненими гранулами поліаміду. Фізико-механічні властивості оцінено за результатами дослідження на розтяг та ударну в’язкість. Показано, що в одержаних композитів високі міцнісні властивості, які зберігаються на рівні ненаповненої матриці. Встановлено, що наявність на поверхні гранул поліаміду мідної оболонки незначно впливає на зміну фізико-механічних властивостей епоксидних композитів. Зроблено спробу пояснити одержані результати з використанням значень міцності адгезійного шару, що формується між епоксидною матрицею і різними за природою поверхнями наповнювача.

Ключові слова: композит; епоксид; поліамід; металізація; мідь.