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The Research of Physical and Mechanical, Thermophysical Properties of Epoxy-Polyester Composite Materials Filled with Discrete Fibers to Increase the Reliability of Vehicles

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Abstract: ***In the article the polymeric materials based on epoxy-polyester binder with addition of dispersed fibers were developed. The mechanical and thermophysical methods were used. The influence of carbon fibers (0.01–0.30 pts.wt.) and cotton (0.01–0.30 pts.wt.) on the physical and mechanical, thermophysical properties of epoxy-polyester composites was investigated. The results of the experiment showed that the introduction of carbon fibers leads to an increase in the fracture stresses, the flexural modulus; the maximum increase was observed for the carbon fiber content q = 0.01 pts.wt. (σ_{fl} = 71.0 MPa, E = 3.8 GPa, W = 7.8 kJ/m^2). The heat resistance of composite materials at this content of carbon fibers is maximal (T = 337 K), and the coefficient of linear thermal expansion (CLTE) is minimal and decreases in all temperature ranges (compared to the matrix). It was found, that the introduction of cotton fibers at a content q = 0.02 pts.wt. into the composition leads to an increase of the flexural modulus from E = 3.6 GPa (matrix) to E = 3.8 GPa and flexural stresses from σ_{fl} = 50.4 MPa to σ_{fl} = 55.2 MPa. The impact strength of such materials decreases from W = 8.3 kJ/m^2 to W = 4.9 kJ/m^2. The results of the study of physical and mechanical properties of composite materials with the addition of cotton fibers were confirmed by thermophysical properties. It was found, that the heat resistance of materials increases from T = 335 K to T = 338 K at this content. The developed composite materials filled can be used to protect equipment, which are exposed to high temperatures or dynamic loads at moderate temperatures.***

Keywords: ***epoxy-polyester matrix, composite material, physical and mechanical properties, thermophysical properties, flexural stresses, flexural modulus, impact strength, heat resistance (by Martens), CLTE, discrete fibers, carbon fiber, cotton, ultrasonic processing, binder, modification.***

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I. INTRODUCTION

At the present stage, the progress of the transport technologies there is a need to employ the polymeric materials in the manufacturing, repair and recovery of separate parts and mechanisms [1]–[4]. This is related to the economic and raw material component of the problem, since the use of such materials reduces the cost of structures and mechanisms, increases their repair period and reliability [5]–[8]. Therefore, the creation of polymeric composite materials (PCMs) with improved properties is relevant today [9]. Particularly, the benefits of PCM over other materials (water resistance, chemical resistance, wear resistance, high adaptability) allow to use them in many industries and to obtain materials with improved performance. In fact, epoxy and polyester resins play an important role among widely used polymers for the development of composite materials (CM) [10]–[13].

The authors [14] studied the physical and mechanical, tribological properties of CM based on epoxy resin. Dependences of influence of the orientation of reinforcing fibers (carbon and glass fibers) on the investigated characteristics was determined. In the paper [15], a PCM, that is characterized by high conductivity and wear resistance based on epoxy resin and graphite, was developed. At the same time, authors [16]–[20] were shown that the CM's physical and mechanical properties were enhanced by influence of energy fields, ultraviolet irradiation, and ultrasonic processing (USP). The use of ultrasonic processing of compositions filled with disperse additives was argued. Properties of polymeric materials based on unsaturated polyester resins of different nature were explored by separately groups of authors: He S., Petkovich ND, Liu K. [21], J. Lin, B. Zhong, Z. Jia [22], Lavoratti A., Scienza LC, Zattera AJ [23] were studied. The advantages and disadvantages of using these binders for the producing of PCM were shown. At the same time, the combination of epoxy and polyester components in one binder allows to combine the benefites of both components and obtain a composite with a new set of properties [24]. The advantages of using multicomponent polymeric materials to improve adhesion, physical and mechanical properties were shown by authors Y. Jahani, M. Ehsani, B. Mottershead, S.J. Eichhorn [25], [26] in their works.

The authors [21] proposed to disperse graphite oxide in unsaturated polyester resin to create nanocomposites. It was found, that fracture energy values increase by 55% with insignificant filler content (0.04 pts.wt.). In the experimental work [23], physical and mechanical, thermophysical properties of composites filled with cellulose nanofibers were studied. It was shown, that the interaction at the phase interface of the fiber/matrix increases with the introduction 1 pts.wt. cellulose fibers and leads to an increase in the properties. It is known, that the use of natural fibers in CM (in particular, which is a waste of industry) allows to increase the operational characteristics and the economic efficiency, which is relevant and advanced. The properties of composite systems at the content of flax [27]–[29], hemp [30], jute [24], ramie [31] and many others have been investigated in various papers. It was shown that the operational characteristics of CM depend on the interphase properties. It should be noted, that the use of carbon fibers and cotton as fillers can solve two problems: the utilization of residues of raw materials from textile production and the possibility of further processing or biodegradation. In the future, the combination of carbon fibers with natural cotton fibers can allow to reduce the number of defects of natural fibers [32]. Moreover, such CM can provide protection of the surfaces of the parts and mechanisms of means of transport, to increase their overhaul resource of life and reliability. Therefore, the development of polymeric materials based on epoxy-polyester binder with addition of dispersed fibers to improve the reliability of sea, river and land vehicles is an urgent task, both material science and the transport industry in general.

II. MATERIALS AND METHODS

For forming a composite matrix with improved physical, mechanical and thermophysical properties the following components were used in the work:

1. A low molecular epoxy diane oligomer ED-20 (q = 100 pts.wt.) was selected as the main component of binder. It should be noted, that the molecules of epoxy oligomers contain glycidyl and epoxy groups, which are capable, interacting with curing agent, to form a cross-linked structure in the materials in a form of a net [33].
2. Ortho-phthalic unsaturated pre-accelerated molding polyester resin Norsodyne O 12335 AL (q = 20 pts.wt. per 100 pts.wt. of epoxy oligomer ED-20).
3. The cold curing agent polyethylene polyamine (q = 10 pts.wt. per 100 pts.wt. of ED-20).
4. The curing agent for polyester resins – Butanox-M50, which is a methyl ethyl ketone peroxide (q = 1 pts.wt. per 100 pts.wt. of polyester resin Norsodyne O 12335).

Discrete carbon fiber and cotton fiber were selected as fillers.

Discrete carbon fiber (dimensions d = 6–8 μm, l = 0.5–1.5 mm) is an artificial fiber which consists of carbon; it is a form of graphite in which carbon atoms are lined into thin long graphite fibers and cotton fiber (dimensions d = 15–25 μm, l = 1.0–1.5 mm) is a textile fiber of vegetable origin.

In order to find the optimum content of fibrous fillers in the epoxy-polyester binder, the physical, mechanical and thermophysical properties of CM were studied. The fiber content was changed within the range of q = 0.01 to 0.30 pts.wt. per 100 pts.wt. of epoxy oligomer ED-20.

In accordance with standards, the following physical and mechanical, thermophysical properties were studied: the flexural modulus, flexural stresses (ASTM D 790 – 03) and the impact strength (ASTM D6110 – 18), heat resistance (by Martens) (ISO 75-2), coefficient of linear thermal expansion (CLTE) (ISO 11359-2).

The technology of forming epoxy-polyester composites was developed in the previous stages of research and presented in [16]. It consists of the following stages: pre-dosing of epoxy diane resin ED-20 and polyester resin Norsodyne O 12335 AL; heating of the resin and fibrous fillers to T = 353 ± 2 K and component exposure at this temperature during time t = 20 ± 0.1 min; the dosage of the filler and the subsequent introduction of it into the binder; hydrodynamic combination of the ED-20 and the fibrous filler during the time t = 1 ± 0.1 min; USP of the composition during the time t = 1.5 ± 0.1 min; cooling the composition to room temperature during the time t = 60 ± 5 min; the introduction of the polyethylene polyamine hardener and mixing the composition during the time t = 5 ± 0,1 min. Further, CM was hardened according to the experimentally ascertained mode: the formation of samples and their holding during the time t = 12.0 ± 0.1 h at a temperature T = 293 ± 2 K, heating at a rate of υ = 3 K / min to a temperature T = 393 ± 2 K, the endurance of the CM during the time t = 2.0 ± 0.05 h, slow cooling to a temperature T = 293 ± 2 K. To stabilize the structural processes in the CM, the samples were kept during the time t = 24 h in air at a temperature T = 293 ± 2 K followed by experimental testing.

III. RESULTS AND DISCUSSION

At first, the dependence of the flexural modulus (E) and the flexural stresses (σ_{fl}) of CM on the carbon fibers content was determined. It was found, that with the introduction of carbon fibers at q = 0.01 pts.wt. the parameters of flexural modulus remain unchanged (Fig. 1, curve 1), but flexural stresses increase from σ_{fl} = 50.4 MPa (matrix) to σ_{fl} = 67.4 MPa (Fig. 2, curve 1). With an additional increase of carbon fibers content (q = 0.02 pts.wt.), we observed a decline of indexes of flexural modulus from E = 3.6 GPa (matrix) to E = 3.3 GPa and flexural stresses to σ_{fl} = 55.2 MPa. At low concentrations, inhomogeneities in the matrix are formed, which destroy the interfacial surface, that is, the adhesion between the fiber and the matrix is reduced. At content q = 0.05 pts.wt. the flexural modulus does not change (compared to the matrix), and the flexural stresses increase to σ_{fl} = 57.2 MPa. The maximum increase of the investigated properties was set at q = 0.10 pts.wt of carbon fibers. The flexural modulus is E = 3.8 GPa, flexural stresses – σ_{fl} = 71.0 MPa. The increase in the elastic properties during flexion can be explained by the high dispersion of carbon fibers during USP, which allows to improve the interaction and adhesion at the interphase "matrix/carbon fiber" separation. In fact, during USP, the process of dispersing and grinding the particles of

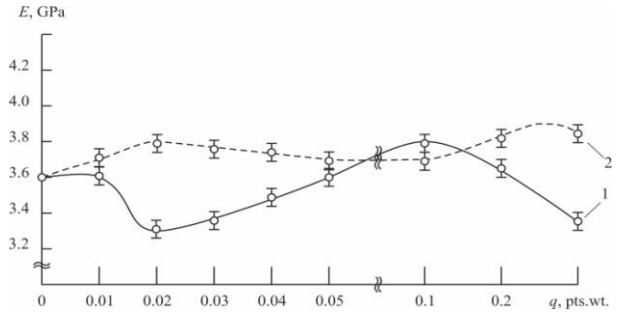

Fig. 1.The dependence of flexural modulus (E) of epoxy-polyester CM on the content of discrete fibers, q, pts.wt.: 1) carbon fiber; 2) cotton fiber.

the filler takes place, which leads to uniform mass transfer of the particles in the epoxy-polyester binder [34]. The increasing of the carbon fiber content within q = 0.10–0.30 pts.wt. leads to a decline of the characteristics to E = 3.5 GPa and σ_{fl} = 62.5 MPa. With increasing concentration above optimal content, the characteristics decreased due to agglomeration of the filler particles destroyed the interfacial interaction. In addition, these agglomerations act as stress concentrators leading to performance decreasing.

On the next step the effect of the cotton fiber particles on the flexural modulus and the flexural stresses were studied. It was determined that the introduction of cotton fibers at q = 0.01 pts.wt. leads to a reduction of the indexes of the fracture stresses from σ_{fl} = 50.4 MPa (matrix) to σ_{fl} = 47.4 MPa (Fig. 2, curve 2). The flexural modulus at this fiber content is almost unchanged and was E = 3.7 GPa (Fig. 1, curve 2). It should be noted, that at q = 0.01 pts.wt. of carbon and cotton fibers the opposite effect of the particles on the properties of the epoxy-polyester matrix was observed (Fig. 2). With a further increasing of the content of cotton fibers in the epoxy-polyester matrix in the range of q = 0.02–0.05 pts.wt. the increase of flexural stresses to σ_{fl} = 52.8–55.2 MPa and stable indexes of flexural modulus E = 3.7–3.8 GPa occurred. By increasing the content of the additive to q = 0.1 pts.wt. the indexes of σfl are reduced to 45.5 MPa, and the flexural modulus was E = 3.7 GPa. With the maximum amount of cotton fibers (q = 0.30 pts.wt.), the fracture stresses have reduced to σ_{fl} = 54.0 MPa (compared to the matrix), and the flexural modulus have raised from E = 3.6 GPa (for the matrix) to E = 3.8 GPa. A slight increase in the elastic properties of CM is related to the mechanism of tensity transfer from the matrix to the fiber [35]. A critical length of fiber significantly affects the stress transfer from the matrix to the discrete fiber according to the Kelly-Tyson model [36]. Since $l \gg d$, it is obvious that the reinforcing effect of the introduction of cotton fibers takes place. Furthermore, due to the chaotic nature of the dispersion of discrete fibers in the matrix, it can be argued, that the maximum stress that the matrix can transmit to the discrete fiber is not reached.

Analyzing the obtained results, it was found that materials filled with carbon fibers at q = 0.10 pts.wt. and cotton fibers at q = 0.02 pts.wt. are characterized by the maximum indexes. However, in the comparative analysis of the dependences' curves of the flexural modulus and flexural stresses on the content of discrete fibers it was found, that CM, filled with carbon fibers at q = 0.10 pts.wt., differ by higher ($\Delta\sigma_{fl}$ = 15.8 MPa) indexes of fracture stresses at the same value of flexural modulus – E = 3.8 GPa.

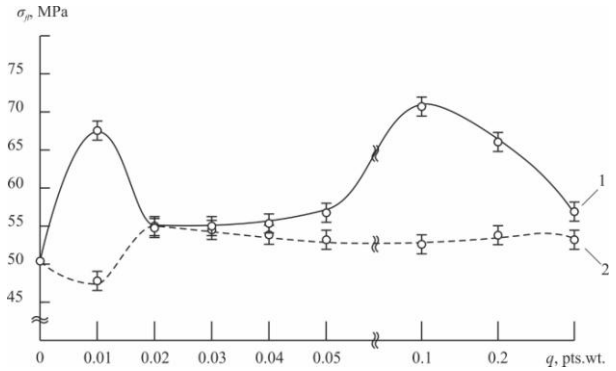

Fig. 2.The dependence of flexural stresses (σ_{fl}) of epoxy-polyester CM on the content of discrete fibers, q, pts.wt.: 1 – carbon fiber; 2 – cotton fiber.

To summarize the physical and mechanical properties, the effect of discrete fibers on the impact strength of epoxy-polyester composites was further investigated (Fig. 3). It was found, that the impact strength decreases from W = 8.3 kJ/m^2 (for the matrix) to W = 6.9 kJ/m^2 at the amount of carbon fibers q = 0.01 pts.wt. At the same content of cotton fibers, the impact strength was reduced to W = 6.1 kJ/m^2. Impact strength of the carbon fiber filled composites increased up to W = 7.9 kJ/m^2 (compared to CM filled with carbon fibers at q = 0.01 pts.wt.) at a loading of q = 0.02 pts.wt. of carbon fiber, presumably due to good dispersion at higher concentrations. Impact strength of CM filled with cotton fibers reduced by ΔW = 3.4 kJ/m^2 (compared to the matrix) at this content. With a further loading of discrete fibers from q = 0.05 to 0.10 pts.wt. the dynamics of impact strength changing for both discrete fibers were the same. Composite materials filled with carbon fibers had the impact strength W = 6.9–7.8 kJ/m^2, and filled with cotton fibers – W = 4.4–5.6 kJ/m^2. The introduction of carbon fibers at critical amount (q = 0.30 pts.wt.) leads to a decreasing of impact strength to W = 7.4 kJ/m^2. The epoxy-polyester matrix, filled with cotton fibers at this content, differs with indexes of the impact strength, which were observed at q = 0.10 pts.wt. – W = 5.7 kJ/m^2 (results are within the error of the experiment).

Therefore, the maximum values of impact strength of the CM, filled with carbon fibers, were observed at q = 0.02 pts.wt. and q = 0.1 pts.wt. However, taking into account the obtained results of the flexural modulus and fracture stresses, it can be stated that the epoxy-polyester matrix at q = 0.1 pts.wt. of carbon fibers is characterized by optimal values. The obtained data characterize CM with such content of carbon fibers as a material with a higher density of crosslinking, in comparison with the epoxy-polyester matrix. For composites filled with cotton fibers, the maximum impact strength was obtained at q = 0.01 pts.wt. (W = 6.1 kJ/m^2). Thus, studies of the impact strength have shown that the destruction of unfilled matrix requires energy more than samples filled with carbon fibers and cotton fibers. That is, if it is necessary to obtain high-impact CM, composite should be formed with high-filled discrete fibers. Therefore, it was believed that the CM was characterized by optimal properties at q = 0.02 pts.wt. of cotton fibers. Since, the flexural modulus and flexural stresses were maximum for such content. Such materials can potentially be used in some applications in

which lightness and good mechanical properties are required, such as automobile or shipbuilding.

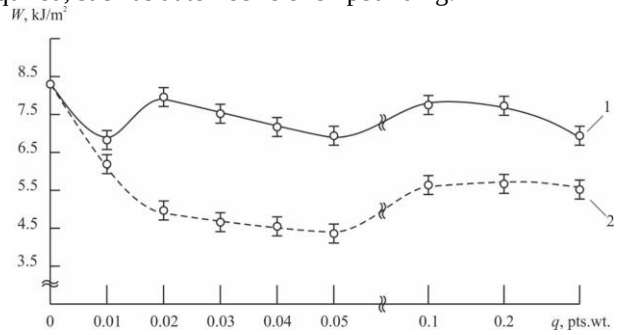

Fig. 3.The dependence of impact strength (W) of epoxy-polyester CM on the content of discrete fibers, q, pts.wt.: 1 – carbon fiber; 2 – cotton fiber.

Fig. 4 shows the dependence of the influence of carbon fibers and cotton fibers on the heat resistance of epoxy-polyester materials. It was determined that the introduction of fibrous fillers at q = 0.02 pts.wt. leads to an increasing of heat resistance indexes from T = 335 K (for the matrix) to T = 337 K (for each type of fiber). With further loading up to q = 0.05 pts.wt. the dynamics of reducing of heat resistance to T = 336 K and T = 334 K (for CM filled with carbon fibers and cotton, respectively) was observed. Heat resistance of composite filled with carbon fibers decreased to T = 333 K, and heat resistance of the CM filled with cotton fibers has not changed and was T = 336 K at q = 0.10 pts.wt. The heat resistance values were also lower at the introduction of discrete fibers at the critical content (q = 0.30 pts.wt.) compared to material filled with q = 0.02 pts.wt. of fibers (Fig. 4, curves 1,2).

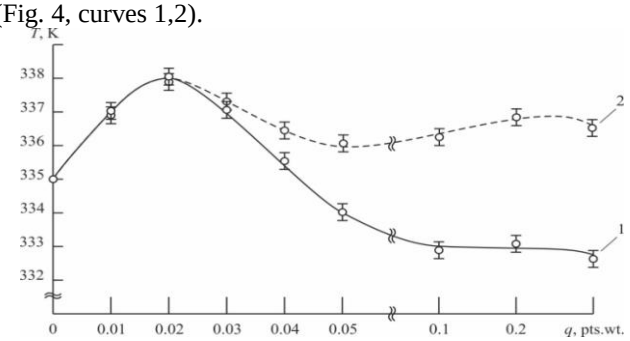

Fig. 4.The dependence of heat resistance (T) of epoxy-polyester CM on the content of discrete fibers, q, pts.wt.: 1 – carbon fiber; 2 – cotton fiber.

The CLTE of epoxy polyester composites filled with discrete fibers was examined in the next stage. The dependencies of thermal expansion of the studied materials in temperature ranges of: ΔT = 303–323 K; ΔT = 303–373 K; ΔT = 303–423 K; ΔT = 303–473 K were determined (Fig. 5, Fig. 6). For the epoxy-polyester matrix, the following CLTE values were obtained: α = 1.6×10-5 K^{-1} (in the region of 303–323 K), α = 2.0×10-5 K^{-1} (303–373 K), α = 3.8×10-5 K^{-1} (303–423 K), α = 8.8×10-5 K^{-1} (303–473 K). It was proved, that the CM characterized by the minimum CLTE values in all ranges at q = 0.01 pts.wt. of carbon fibers: in range of 303–323 K – α = 1.4×10-5 K^{-1}, in range of 303–373 K – α = 2.0×10-5 K^{-1}, in range of 303–423 K – α = 3.7×10-5 K^{-1}, in range of 303–473 K – α = 8.4×10-5 K^{-1}. The dynamics of a slight increasing of CLTE values in temperature ranges of 303–323 K, 303–373 K (Fig. 5, curves 1,2) were observed at q = 0.02–0.10 pts.wt. of carbon fibers It should be noted, that the epoxy-polyester materials are also characterized by an increasing of CLTE indexes: α = (4.4–4.9)×10-5 K-1 and α = (9.5–9.7)×10-5 K^{-1} in the temperature ranges ΔT = 303–423 K, ΔT = 303–473 K, respectively. The dynamics of the decrease in CLTE values was observed at q = 0.30 pts.wt. of carbon fibers. However, during a comparative analysis of the physical and mechanical and thermophysical properties of composite materials filled with carbon fibers, it was found, that the composite, filled with q = 0.10 pts.wt. of additive, is characterized by the optimum properties in the complex.

It should be noted, that carbon fibers dispersion at low content in the polymer resin can potentially elevate matrix properties of such carbon fiber filled composites. But, instead of having a positive effect on CLTE, dispersion of discrete carbon fibers was resulted in an insignificant decline in CLTE, possibly due to a more non-homogeneous distribution of discrete carbon fibers in the epoxy-polyester binder as a result of filtering effects by the carbon fibers [35]. The obtained results are in agreement with the work executed by Inam et al. [35].

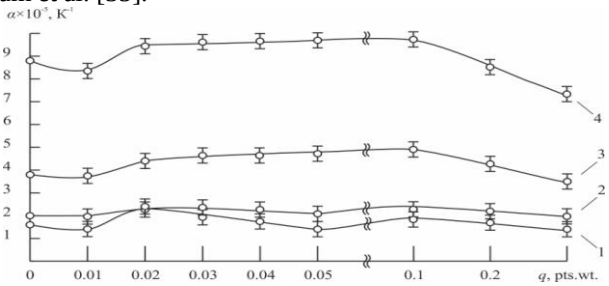

Fig. 5.The dependence of coefficient of linear thermal expansion on carbon fiber content at different temperature ranges: 1 – ΔT = 303–323 K; 2 – ΔT = 303–373 K; 3 – ΔT = 303–423 K; 4 – ΔT = 303–473 K.

The study results of CLTE of the materials, filled with cotton fibers, are shown in Fig. 6. The loading of cotton fibers at q = 0.01 pts.wt. leads to increasing of CLTE values in all temperature ranges: by Δα = 1.2×10-5 K^{-1} in range of 303–323 K, Δα = 0.5×10-5 K^{-1} in range of 303–373 K, Δα = 1.1×10-5 K^{-1} in range of 303–423 K, Δα = 0.9×10-5 K^{-1} in range of 303–473 K (compared to epoxy-polyester matrix). The dynamics of decrease in CLTE values in all temperature ranges was observed at q = 0.02 pts.wt. of additive: α = 1.7×10-5 K^{-1} in range of 303–323 K, α = 2.0×10-5 K^{-1} in range of 303–373 K, α = 4.4×10-5 K^{-1} in range of 303–423 K, α = 9.3×10-5 K^{-1} in range of 303–473 K. In this case, the CLTE values in ranges 303–323 K and 303–373 K are at the equal level with the epoxy-polyester matrix. It was found, that the CLTE values remained constant within the limits of the error of the experiment (Fig. 6, curves 1–4) with the further increasing of the content of cotton fibers in the range q = 0.05–0.30 pts.wt. (until the critical value was reached). Taking into account the values of heat resistance for materials filled with cotton fibers, it is determined that the composite at q = 0.02 pts.wt. of cotton fibers was characterized by the optimal indexes of thermophysical properties. The obtained results correlate with the results of the study of the physical and mechanical properties of such CM. The increase in heat resistance and reducing CLTE of CM filled with cotton fibers (85-90% cellulose) is due to the fact that cellulose particles have reduced thermal expansion and good dimensional stability.

It is obvious, that even with insignificant fiber content, due to their chaotic dispersion by USP, an increase in thermophysical properties of the studied CM was achieved. Due to the thermal expansion discrepancy between cotton fibers and the epoxy-polymer binder, residual stresses are supposed to be tensile in the polymeric phase and compressive in the cotton fibers, and during heating, the residual stresses relaxed elastically or plastically [28].

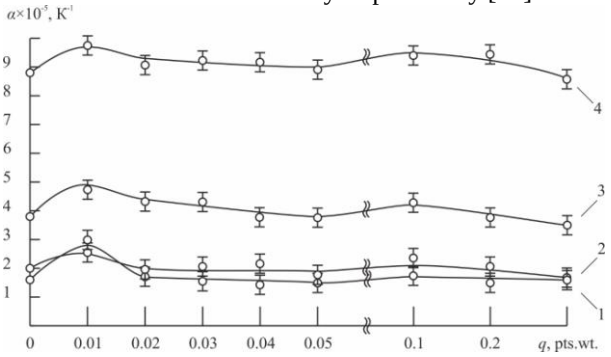

Fig. 6.The dependence of the coefficient of linear thermal expansion on the content of cotton fibers at different temperature ranges: 1 – ΔT = 303–323 K; 2 – ΔT = 303–373 K; 3 – ΔT = 303–423 K; 4 – ΔT = 303–473 K.

During a comparative analysis of the thermophysical properties of CM with two different discrete additives, it was found, that epoxy-polyester matrix filled with cotton fibers had lower CLTE and heat resistance (compared to a matrix filled with carbon fibers). At the same time, the material filled with q = 0.10 pts.wt. of carbon fibers was characterized by the increased physical and mechanical properties. Therefore, it was considered that the composite materials filled with q = 0.02 pts.wt. of cotton fibers is better to use to protect equipment, which is exposed to high temperatures, and coating, at q = 0.10 pts.wt. of carbon fiber – to protect the parts and equipment that are exposed to dynamic loads at moderate temperatures.

IV. CONCLUSIONS

Physical and mechanical and thermophysical properties of epoxy-polyester materials filled with carbon and cotton fibers, were investigated. According to the results of experimental study, the following can be stated:

1. It was determined, that the composites, filled with q = 0.10 pts.wt. of carbon fibers, were characterized by increase in physical and mechanical properties. It was proved, that the flexural modulus of the developed materials increased from E = 3.6 GPa (for epoxy-polyester matrix) to E = 3.8 GPa, flexural stresses increased from σ_{fl} = 50.4 MPa to σ_{fl} = 71.0 MPa, and the impact strength was slightly reduced from W = 8.3 kJ/m^2 to W = 7.8 kJ/m^2.

2. It was found, that the introduction of discrete carbon fibers at q = 0.01 pts.wt. leads to a slight increasing of the heat resistance (by Martens) from T = 335 K to T = 337 K. For such carbon fiber content, the CLTE indexes at all temperature ranges are: in ΔT = 303–323 K – α = 1.4×10-5 K^{-1}, in ΔT = 303–373 K – α = 2.0×10-5 K^{-1}, in ΔT = 303–423 K – α = 3.7×10-5 K^{-1}, in ΔT = 303–473 K – α = 8.4×10-5 K^{-1}.

3. It was analyzed, that the values of physical and mechanical properties of composite materials increases at q = 0.02 pts.wt of cotton fibers. In this case, the values of flexural modulus increase from E = 3.6 GPa to E = 3.8 GPa, the flexural stresses - from σ_{fl} = 50.4 MPa to σ_{fl} = 55.2 MPa. However, the impact strength of such materials decreases from W = 8.3 kJ/m^2 to W = 4.9 kJ/m^2.

4. It was additionally determined, that the obtained results of the study of thermophysical properties of CM correlate with the data of the investigation of cohesive properties. Optimal heat resistance values were observed at q = 0.02 pts.wt. of cotton fibers – T = 338 K. In this case, the values of the coefficient of linear thermal expansion in all studied ranges were reduced in comparison to the epoxy-polyester matrix: in range of 303–323 K – α = 1.7×10-5 K^{-1}, in range of 303–373 K – α = 2.0×10-5 K^{-1}, in range of 303–423 K – α = 4.4×10-5 K^{-1}, in range of 303–473 K – α = 9.3×10-5 K^{-1}.

It should be noted, that the epoxy-polyester matrix filled with q = 0.02 pts.wt. of discrete cotton fibers is appropriate to use for protection of equipment that are exposed to alternating temperatures, and polymer coating filled with q = 0.10 pts.wt. of carbon fibers – for protection against dynamic loads at moderate temperatures.

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