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EFFECT OF INCREASED TEMPERATURES ON THE DEFORMATION AND STRENGTH CHARACTERISTICS OF A GFRP BASED ON A FABRIC OF VOLUMETRIC WEAVE

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The aim of this research was to experimentally study the property degradation of a glass-fiber plastic with increasing temperature and the features of its mechanical behavior. A glass-fiber plastic of aerospace design based on a fabric of volumetric weave from a K11C6170-BA silica thread and an EDT-10P epoxy binder was investigated. The results of tensile tests performed using a technique harmonized with domestic and foreign methods for testing GFRP specimens at temperatures of 22, 100, 175, 250, and 300ºC are presented. Their deformation diagrams are obtained, and values of the basic mechanical characteristics are determined. Relations describing the degradation of the elasticity modulus, the tensile strength, Poisson ratio, and the strains corresponding to the strength limit with increasing temperature are constructed and analyzed. Features of the mechanical behavior of the composite material are discussed; in particular, the nonlinear change in strength and qualitative changes in the types of deformation diagrams with growing temperature, which are associated with changes in deformation and fracture mechanisms. An analysis of degradation of glass-fiber plastic structures after the tests at normal and increased temperatures is performed based on data found with the use of a stereomicroscope.

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Introduction

The wide application of composite materials in various branches of industry is accompanied by an equally rapid development of technologies and the scientific and technical base for designing products from them [1]. One of the most claimed classes of composites are polymer composite materials for structural purposes — as a rule, in the form textolites and laminates [2, 3]. Laminates and textolites have one obvious drawback — a low interlaminar strength. The modern methods for combating this drawback is stitching fabrics and the use of fabrics of volumetric weaving.

Multilayered textile materials are used as three-dimensional spatially reinforcing fillers for highly effective composite materials in such areas as aerospace industry, mechanical engineering, aircraft production, shipbuilding, metallurgy, heat-and-p0wer engineering, and atomic industry. Multilayered glass fabrics of volumetric weaving are used as reinforcing materials for of the glass-fiber plastic working under complex and severe conditions with action of high-speed aerodynamic streams and vibrations at high temperatures. In such cases, the basic advantage of multilayered fabrics — their impressive strength in the transverse (perpendicular to layers) direction, preventing these glass-fiber plastics from delamination during their operation, is realized. This cannot be achieved by other kinds of reinforcing materials. Reviews of the technological and structural aspects and the current status and prospects for using 3D fabrics in polymer composites are presented in [4, 5]. In [4], problems and advantages 3D woven structures are considered, and a conclusion about the overwhelming advantages of the so-called multinuclear weaving is made. In [5], questions about the use of various 3D-reinforced polymer composites in technical applications are discussed. Study [6] is devoted to advanced technologies of producing 3D-reinforced composites with adapted orientations of fibers and integrated monitoring systems based on optical fiber sensors in automated manufacture.

In [7, 8], a comprehensive comparative experimental investigation was performed into the behavior of an E-glass-fiber composite in tension in its plane. The composite consisted of two woven 3D orthogonal monolayers made by the VARTM technology with the use of 3WEAVE® perform and a laminate reinforced with four layers of E-glass fabric of plane weave. The results obtained showed that the 3D nonblooming orthogonal textile composites had considerably higher ultimate failure stresses and strains in their planes and higher strain thresholds of damage initiation than their multilayered composite analogues.

In using polymer composites for producing critical structures, a topical problem is the analysis of influence of elevated and reduced operation temperatures on the mechanical properties and destruction mechanisms of such materials. Experimental data about the effect of operation and climatic temperatures on the mechanical properties of different classes of polymer composite materials are presented in [9-15]. In [15-17], and temperature dependences of the mechanical properties of composites are analyzed. In [18-21], attempts are made to model the thermoelastic characteristics of composites, including spatially reinforced polymer plastics [21]. In [22], a nontrivial dependence is found to exist for the loss modulus as a function of temperature T, with an extremum T = 130-150ºC for a pultruded E-glass-fiber composite with a polyester binder. However, for a 3D glass-fiber plastic with an epoxy binder, the relations of mechanical characteristics in three-point bending had no specific features [23]. In [24], relations for the strength and stiffness characteristics of a three-dimensional four-directional carbon plastic with an epoxy binder as functions of temperature and aging time are presented. A monotone decrease in its characteristics with growing aging time and base temperature is noted. However, at T = 90ºC, a growth in aging time increased its elastic modulus.

Thus, for 3D-reinforced structures in a polymeric matrix, especially important become problems on establishing temperature dependences for the elastic and strength characteristics of fibrous composites. In the given work, multilayered silica fabrics (MKT) made from silica yarns of mark K11S6-170BA, preliminary subjected to texturing (bloating), were used. The advantages of the composites made on the basis of these materials were as follows: delamination and crack localization safety; high resistance to end impacts; increased resistance to deformation loadings and high-temperature actions; ablation resistance and radiotransparency; reduced cost and labor input in manufacturing highly effective composites; the shape

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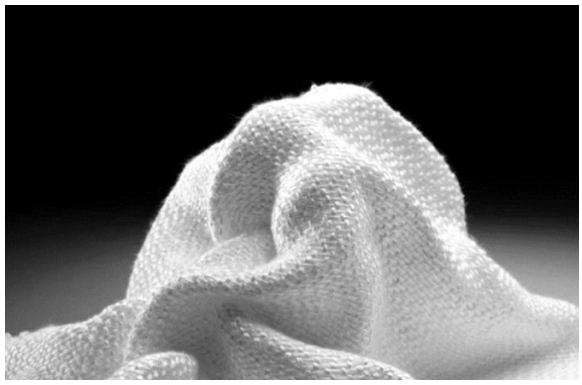

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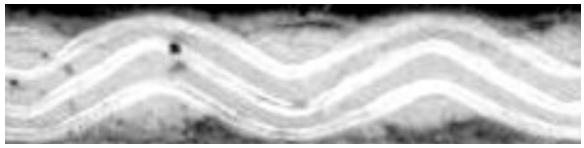

Fig. 1. Appearance of a multilayered MKT-3.0 silica fabric (a) and glass-fiber plastic (from the end) on the basis of MKT-3.0 and EDN-10P (b).

of preform practically agrees with that prescribed for the composite and needs minimum processing after impregnation; improved reproducibility of the manufacture process. As a binder, the widespread and inexpensive EDT-10P was used.

The purpose of the present work was to investigate the degradation of mechanical characteristics of glass-fiber plastics on the basis of a MKT-3.0 fabric of volumetric weaving , made from fire-resistant K11S6170-BA silica yarns and the EDT-10P epoxy binder at growing temperature. In our work, results of experimental investigations into the influence of temperatures on the deformation and strength properties of the glass-fiber plastics in tension are presented.

1. Materials and testing equipment and techniques

Investigated was a glass-fiber plastics of aerospace purpose on the basis of a multilayered MKT-3.0 fabric of volumetric weaving (3.0 is the running mass of the fabric, kg/m^2) made from K11S6170-BA silica yarns and the EDT-10P epoxy binder. MKT had a fusion temperature of up to 1400°C, and shrinkage of the woven fabric at 1000°C did not exceed 1%. Appearance of the fabric is shown in Fig. 1.

The EDT-10P epoxy used as the matrix had a density of 1200-1300 kg/m^3 and heat resistance of 110°C on Martens scale. In appearance, it was a homogeneous viscous paste of light yellow color, without extraneous inclusions.

The physical characteristics of glass-fiber plastics made from MKT-3.0 and EDT-10P (Fig. 1b) were as follows: binder 33.8 wt.%, degree of cure of binder 91.6%, density 1.67 g/cm^3, and plate thickness 3.5-4.0 mm.

Tensile tests of the composite were performed at T = 22, 100, 175, 250, and 300ºC at the CKP of the Center of Experimental Mechanics PNIPU on an Instron 5882 universal electromechanical test system equipped with a temperature chamber with working temperatures from –100 to 350ºC. In total, 19 samples were tested.

The longitudinal deformations of the samples were measured by an AVE Instron 2663-821, videoextensometer, whose operation is based on determining the coordinates of contrasting (white or black) labels of the measuring base, marked the working part of sample (Fig. 2), by means of a digital high-resolution videocamera. The field of vision of the

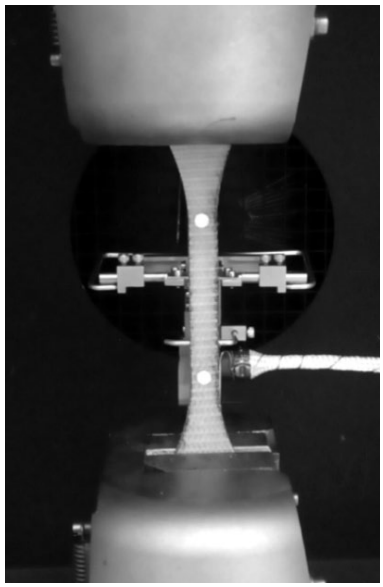

Fig. 2. Sample with white labels for measuring the longitudinal deformations and an extensometer of transverse deformations in tensile tests.

videoextensometer is 200 mm, the digitization speed of videosignal — one frame of information per 20 μs, and the absolute error of measurements — 2 μm. The transverse deformations were measured by Epsilon 3575-250M-HT2 (at 22 and 100ºC) and Epsilon 3675-025M (at 175, 250, and 300ºC) extensometers with an accuracy of 0.2% (see Fig. 2).

The tensile tests were carried out according to the GOST 25.601-80 recommendations relating to the form and geometry of samples, thermostating at elevated temperatures, loading rate, and the statistical processing of results. In using the given technique, the mistakes of the standard described in [25], in particular, those connected with calculations of mechanical characteristics, were taken into account. The samples had the form of a flat dumbbell, of general dimensions 235 × 25 × 3 mm. Their working part was 70 mm long and 18 mm wide, with transition radii ≥ 80 mm. The labels for measuring deformations by the videoextensometer were marked in the working part of samples (see Fig. 2). The ultimate strength was determined as the ratio of the maximum load to the cross-sectional area of the working part of sample, the elastic modulus — as the ratio of stress to the corresponding strain in the interval from 0.1 to 0.3%, and the Poisson ratio — as the ratio between transverse and longitudinal strains.

The crosshead motion speed was 2 mm/min. Before tests at the temperatures of 100, 175, 250, and 300ºC, the samples were thermostated. This procedure included the linear heating of samples to the chosen temperature at a rate of 10 ºC/min and holding them all for 2 h and each sample for 0.5 h after its installation.

We should note that the temperatures above 110ºC are not operating ones for the given composite, but this material is used in products and structures in which, in the case of nonstandard and emergency situations, temperatures can reach 300ºC and more. Thus, in the design and choice of materials, the information on the residual properties and serviceability of composites in postlimiting modes is necessary and topical.

An analysis of structural degradation of the glass-fiber plastic after tests at normal and elevated temperatures was carried out on the basis of data obtained with the help of a 30-power Zeiss STEREO Discovery. V12 stereomicroscope.

TABLE 1. Results of Tensile Tests on Glass-Fiber Plastics at T = 22, 100, 175, 250, and 300ºC

Sample number	E, GPa	σ_b, MPa	ε_σ, %	ν	T, ºC
1	10.8	119	2.89	0.10	22
2	11.3	132	3.35	0.18	
3	10.7	114	3.05	0.18	
4	10.5	126	3.05	0.10	
5	9.5	107	3.08	0.08	
Average value	10.6±0.7	120.0±10.0	3.08±0.17	0.13±0.05	
6	2.0	74	5.28	0.17	100
7	1.7	73	5.46	0.13	
8	2.2	78	4.79	0.19	
9	2.1	76	5.07	0.18	
10	2.0	77	5.20	0.16	
Average value	2.0±0.2	76.0±2.0	5.16±0.25	0.17±0.02	
11	1.7	70	5.80	0.11	175
12	1.6	75	6.80	0.16	
Average value	1.65***	72.5***	6.3***	0.14***	
13	1.3	64	7.48	0.09	250
14	1.4	72	7.51	0.18	
15	1.2	58	8.53	0.09	
16*	1.9	64	-*	0.11	
17	1.3	58	8.64	0.24	
Average value	1.3±0.1	63.0±6.0	8.04±0.63	0.14±0.07	
18	0.8	88	10.32	0.16	300
19	0.9	99	9.90	-**	
Average value	0.85***	93.5***	10.11***	0.16[4*]	

*It was impossible to establish the values of deformation at the maximum stress

**Transverse deformations were not recorded during loading

***The average of values; the root-mean-square deviation was not calculated

[4*]Value for one sample.

2. Results of tests

From the results of tensile tests on glass-fiber plastic samples, the maximum load N_{max}, strength σ_b, elastic modulus E, the strain ε_σ corresponding to the strength σ_b, and Poisson ratio ν were determined.

Five samples were tested at each basic temperature (22, 100, and 250ºC) and two samples at each additional temperature (175 and 300 ºC). Results of the tests are shown in Table 1 and Fig. 3.

The statistical processing of the experimental data found at the basic temperatures was carried out according to GOST 25.601-80, but for those at additional temperatures — the arithmetic mean or one value was taken.

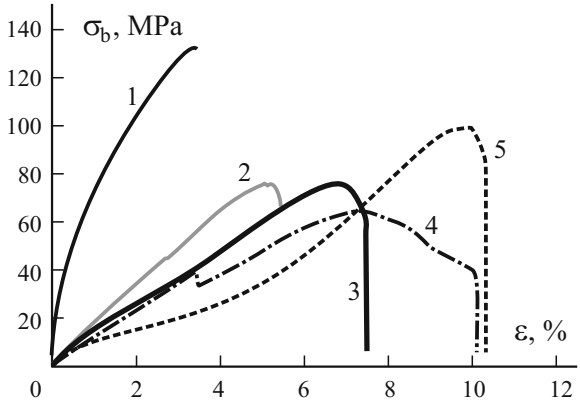

Fig. 3. Deformation diagrams of glass-fiber plastic samples at T = 22 (1), 100 (2), 175 (3), 250 (4), and 300 ºC (5).

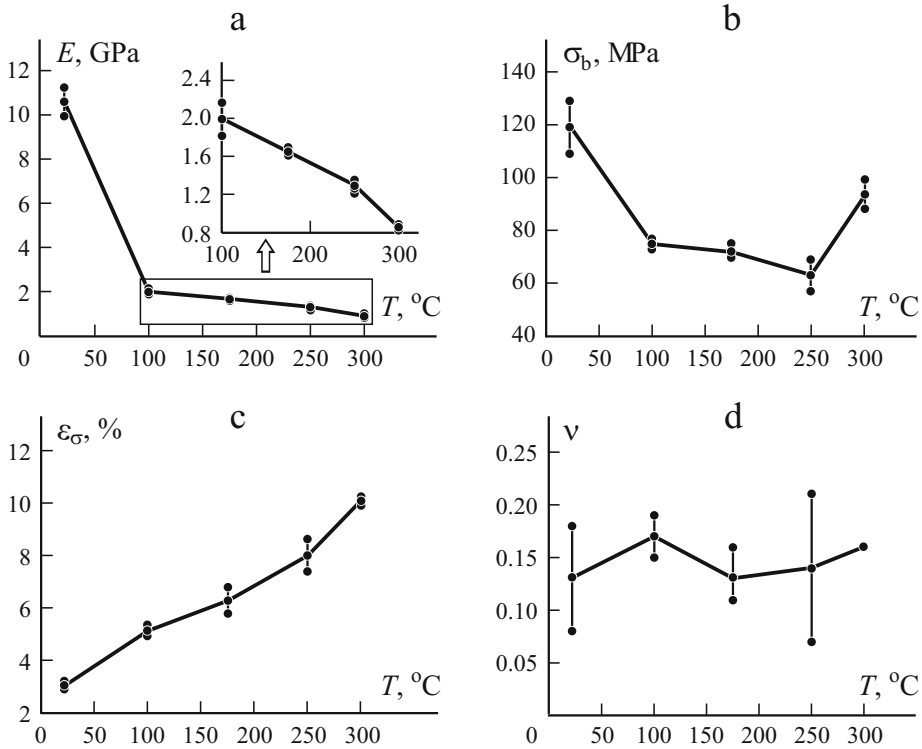

Fig. 4. The elastic modulus E (a), ultimate strength σ_b (b), strain ε_σ corresponding to the ultimate strength (c), and Poisson ratio ν (d) of glass-fiber plastic vs. T.

3. Discussion of results

At temperatures increasing from 22 to 100ºC, the elastic modulus (see Fig. 4a) decreased 5-6 times (from 10.6 to 2.0 GPa), but at temperatures from 100 to 300ºC — more than twofold (from 2.0 to 0.85 GPa). This was caused by softening of the EDT-10P matrix.

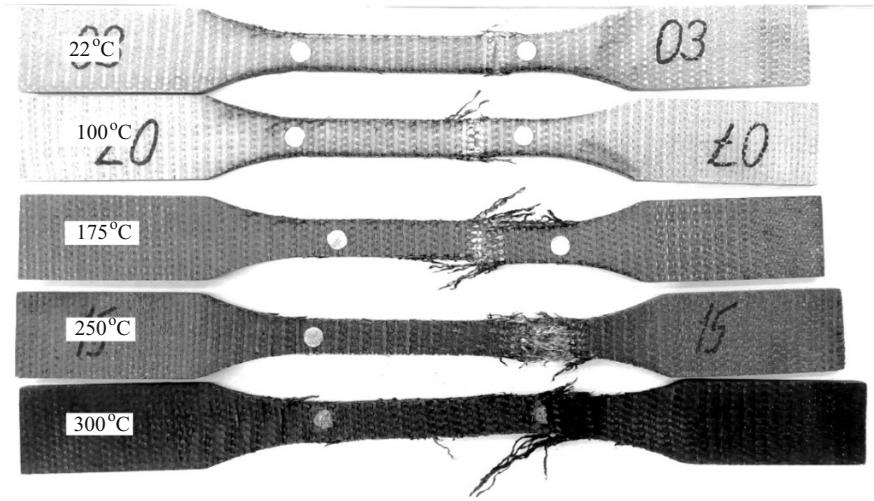

Fig. 5. View of rupture and magnitude of irreversible deformation of glass-fiber plastic samples at different temperatures.

The ultimate strength in the range from room temperature to 100ºC decreased by 37% (from 120 to 76 MPa), but from 100 to 250ºC — by 17% (from 76 to 63 MPa). On the following temperature interval, from 250 to 300ºC, an interesting effect was observed — the average strength grew by 48% (from 63 to 93 MPa) (see Fig. 4b).

This effect can be explained by the fact that, at such high temperatures, the binder was seriously destructed, the low temperature carbonation of the polymer in air occurred, its rigidity grew considerably, and the matrix-fiber system operated differently than at lower temperatures. This destruction led to the rise of distinctive rigid hinges in the loopy rows of reinforcing material on the one hand and to the crumbling of matrix material and occurrence of voids in interbraid and interfiber spaces on the other hand. Thus, the fiber braids became able to unfold in the loading direction and to participate in operation on the rigid hinges. Both these processes increased the number of the fibers operating in the loading direction, as a result of which the ultimate strength grew.

Indirectly, this is also confirmed by character of the deformation diagrams found in tests at 300ºC (see Fig. 3). Their shape rather resemble the deformation of "dry" fabrics than of a composite material. The diagrams have a characteristic initial section which, for fabrics and textile materials, are interpreted as the stage of untwisting and straightening of interlacing yarns (fibers) [26].

The temperature dependence of the strain corresponding to the ultimate strength is depicted in Fig. 4c. It is seen that the strain grew quasi-linearly from 3 to 10% as the temperature increased from 22 to 300ºC.

The Poisson ratio (see Fig. 4d) did not exhibit distinct variations with growing temperature in view of a huge statistical scatter of experimental data. This scatter can be explained by the highly inhomogeneous structure and, accordingly, the geometry of sample surfaces, to which the strain gage had been fastened. Despite this fact, it can be concluded that the Poisson ratio varied between 0.1 and 0.16.

On Fig. 5, the appearance of glass-fiber plastic samples after rupture at normal and elevated (100, 175, 250, and 300ºC) temperatures are show. It can be noticed that, with rising temperature, their relative lengthening at rupture grew increasingly.

To check the noted increase in the ultimate strength, the surface structure of samples outside the rupture zone was analyzed. The results obtained with the help of an optical stereomicroscope confirmed that, with rise in temperature, a net of erosive voids had evolved around fiber braids (Fig. 6). The darkening of the material points to the carboning of polymer. Thus, at thermostating the composite at 300ºC in air, the destruction and carbonation of polymer reached such a level that

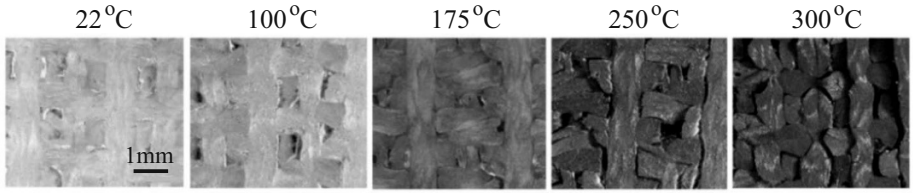

Fig. 6. Development of destruction of binder at increasing surface temperature of a glass-fiber plastic at a 65× magnification.

the system of interlaced fibers could turn into the loading direction, and the curved fiber braids around of the rigid hinges in the matrix were included in the work. Ultimately, this resulted in smoothed stress fields and in an increased strength.

We should note that tests at 500ºC were also designed, but already at 410ºC, the sample ignited, and the tests were recognized impossible.

Conclusions

As a result of our research, the mechanical characteristics of a composite material on the basis of an MKT-3.0 fabric of volumetric weaving from a fire-resistant K11S6170-BA silica thread and an EDT-10P epoxy binder were determined. Tensile tests have been performed, and temperature dependences of these characteristics at temperatures from 23 to 300ºC were constructed.

The elastic modulus (see Fig. 4a) was found to decrease 5-6 times (from 10.6 to 2.0 GPa) as the temperature grew from 22 to 100ºC, and more than twice (from 2.0 to 0.85 GPa) at temperatures from 100 to 300ºC.

The ultimate strength of the composite increased (see Fig. 4) as the temperature grew from 250 to 300ºC and became by 50% higher than the minimum one and only by 25% lower than the strength at 22 ºC (see Fig. 4b). In authors' opinion, such an effect is typical only of spatially reinforced composites. It becomes especially curious in view of that fact that deformation characteristics of the material grew monotonically and the elastic modulus decreased monotonically.

The Poisson ratio (see Fig. 4d) varied irregularly between 0.1 and 0.16) and had a high statistical scatter.

During tests, self-ignition of the composite in air at T = 410ºC was also fixed.

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