Experimental Characterization of Composite Material Properties

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Abstract For determination of actual mechanical properties of composite material used in the manufacture of natural samples the original technique was proposed. Experimental characterization of composite material, intended for the repair of damaged pipelines during optimization of its service characteristics, revealed that introduction of disperse fillers in epoxy resin had a positive effect on the mechanical properties. Intensive experimental study showed that concentration of montmorillonite nanofiller had a significant impact on dynamic modulus of elasticity of composite materials based on epoxide resin while application of perforated metal tape in polymer resin composites demonstrated increasing of composite mechanical strength.

Keywords Composite material \cdot Tensile test \cdot Disperse filler \cdot Dynamic modulus \cdot Perforated metal tape

1 Introduction

Investigations in composite materials properties in the course of project implementation were carried out mainly for:

 determination of actual mechanical properties of the materials used in the manufacture of natural samples with bandage for using them in calculation of the bandage geometric parameters;

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(ii) optimization of service characteristics of composite materials used for the repair of damaged pipelines in view of features of subsequent operation.

2 Actual Mechanical Properties

2.1 Materials of Reinforcing Element and Resin

As a reinforcing element during preparation of full-scale specimens I3 and I4 was used straight roving (Fig. 1) from fiberglass EC10 1680N-U10(168) [1], where in designation E—general purpose glass; C—continuous thread; 10—nominal diameter of elementary filament, μ m; 1680—nominal linear density, teks; *N*—for winding; U10—lubricant; 168—linear density of complex threads, teks. Single strand of roving consisted of ~ 1200 filaments. The roving was used in combination with epoxy resin KDA-HI [2].

Strength characteristics of the polymer matrix according to manufacturer's information after standard mode curing (100–180 °C during 2 h):

- flexural strength—90–110 MPa;
- tensile strength—75–80 MPa;
- impact strength—5–10 kJ/mm²;
- elongation—5.5–8%.

2.2 Test Samples of Composite Material

To determine the mechanical properties of the composite material, the samples in the form of loop were manufactured. Specimen consisted of two branches, a and b (Fig. 2a). The loop formed by winding of the roving, soaked with epoxy, on special rig (Fig. 2c). During the winding the roving was stretched by force, which value was varied (Table 1).



Fig. 1 Roving used as reinforcing element



Fig. 2 Tensile sample: **a** geometrical dimensions: *asterisk* the same dimensions for all samples; <> average size; *a*, *b*, *c*, *d*, *e*, *f*, *g*, *h* sections of sample; **b** sample before test; **c** rig for samples preparation

Parameter		Section	Sample					
			P1	P2	P3	P4	P5	
Preliminary tension of roving N_1 , kg ^a			2	4	3	3.5	0.1	
Number of roving turns			80	80	80	80	75	
Width, mm Bran Rour	Branch	a	7.3	6.8	6.7	7.0	8.0	
		b	7.1	6.8	6.5	7.0	8.6	
	Rounding	c	4.9	5.1	5.3	4.9	5.2	
		d	4.9	5.0	4.9	5.2	5.5	
		e	4.8	4.6	4.8	5.0	5.0	
		g	4.9	4.7	4.7	4.9	5.0	
		f	4.9	4.7	5	4.8	5.0	
		h	4.9	4.6	4.7	5.0	5.5	
Location of fracture in section			g	f	e	e	g	

Table 1 Characteristics of tensile samples

^aAmplitude variation during winding ± 1.5 kg

2.3 Test Results of the Composite Material

The test was carried out on machine Instron 8802 using biaxial extensiometer 2620-614 with longitudinal and transverse base of measurement (25 and 15 mm, respectively, Fig. 3). The speed of deformation was 10 mm/min.

Figure 4 gives details of the sample P2 tension. Linear dependence between tensile load and force is true in all range of deformation up to the sample break.

Summary of the test results of specimens P1–P5, brought to single roving, are present in Table 2. It should be noted that dependence of the destructive force and ultimate deformation on preliminary tension of roving was not detected.

2.4 Tensile Test of the Roving

The test was carried out on machine Instron 8802 equipped with snail grips (Fig. 5a) and extensioneter 2620-601. The sections of the roving, spooled from reel,



Fig. 3 Tensile test: a sample in grips; b features of samples fracture



Fig. 4 Tension of sample P2

Sample→	P1	P2	P3	P4	P5	Average
Preliminary tension of roving (N)	19.6	39.2	29.4	34.3	1	24.7
Fracture force (kN)	95.28	101.23	99.19	96.04	99.87	98.32
Maximal strain (%)	1.36	1.44	1.59	1.39	1.58	1.47
Poisson's ratio (µ)	0.179	0.196	0.164	-	0147	0.172
Young's modulus ^a E (MPa)	32,721	34,645	31,673	33,079	25,518	31,527
Tensile strength ^a (MPa)	445.0	498.9	503.6	459.8	403.2	462.1

Table 2 Results of tensile tests

^aThickness in the middle part of the loop was used in the calculation of the mechanical properties of composite

were used as samples with next parameters: measuring base is 52.5 mm, length of working section is 140 mm, speed of displacement during test is 30 mm/min.

Test diagrams of samples are present in Fig. 5b. Comparison of test results of the roving and composite material is given in Table 3.

2.5 Additional Research of the Material

2.5.1 Linear Density of the Roving

A sample of roving with length of 100 ± 1 mm was weighed on Denver Instrument APX-60 (maximum permissible weight is 60 g, measuring accuracy is 0.1 mg) (Table 4).



Fig. 5 Tensile test of roving: a general view of the roving test; b tensile diagram: *R1, R2, R3* Samples marking; *1* Approximation of diagrams with linear dependence

Roving			Composite material			
Sample	ē	F_1, H	Sample	ē	F_1, H	
R1	0.0164	620	P1	0.0133	595.5	
R2	0.0131	690	P2	0.0142	632.7	
R3	0.0170	600	P3	0.0144	620.0	
			P4	0.0124	600.3	
			P5	0.0159	665.8	
Average	0.0155	636.7	Average	0.0140	622.9	
min	0.0131	600	min	0.0124	595.5	
max	0.0170	690	max	0.0159	665.8	

Table 3 Test results of the roving and composite material

The difference in linear density of roving, according to measurements is 1721.4 teks and certificate is 1760 teks was $\sim 2.2\%$.

2.5.2 Photomicrography of the Roving

Based on pictures (Fig. 6), average filament diameter was $\sim 10 \ \mu m$.

2.5.3 Measurement of the Cross-Section Area of Roving

After dense packing of 57 roving strands in a groove under tension force of ~ 50 N, the calculated cross-section area of 1 roving amounted to 1.044 mm².

Note that average area per 1 roving of samples P1–P5 was: for sections (a, b) 1.36 mm²; (c, d) 0.96 mm²; (e, f, g, h) 0.92 mm², respectively (Fig. 6).

Table 4	Weight	of rov	ing with	1 m	length
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No. weighing	1	2	3	4	5	6	7	8	9	Average
Weight (g)	1.7214	1.7215	1.7212	1.7214	1.7215	1.7214	1.7214	1.7214	1.7215	1.7214

Fig. 6 Filaments of roving



3 Optimization of Service Characteristics

3.1 Effect of Disperse Fillers

The effect of disperse fillers including nanoparticles on the mechanical properties of epoxy based composites for the repair of pipelines using composite-muff technology has been studied [3]. Epoxy resins of different grades (ED-20 and E-181) have been used as binders for composite materials. As the disperse fillers the following materials have been used: metal powder, carbonyl iron, graphite, calcium stearate, and various epoxy-silicates of metals. The composites have been prepared by mixing all components at low shear. The results demonstrated that an introduction of disperse fillers in epoxy resin had a positive effect on the mechanical properties of examined composites.

The plot, shown in Fig. 7a, proves that the elasticity modulus of the composite materials increases by 5-15%. The plot in Fig. 7b proves that material yield strength increases by 16-35% in dependence on the filler type. Analysis of mechanical fracture pattern shows that the introduction of fillers leads to embrittlement of the composite material as compared to the initial resins. This causes the need for the introduction of additional plasticizers of the matrix phase.



Fig. 7 Effect of the disperse fillers on the elasticity modulus of the composite (a) and yield strength (b) of epoxy-based composites (1-graphite (GKZ) + calcium stearate, 2-metal powder (Fe), 3-carbonyl iron (VKZ), 4-graphite (GKZ) + epoxy silicate Cu + cal

3.2 Dynamic Mechanical Characteristics

An experimental study of the dynamic mechanical characteristics of composite materials based on epoxide resins and intended for a composite-sleeve pipeline technology has been performed. The aim of the investigation was to estimate an efficiency of their application in raising resistance of the pipelines to hydraulic shock and vibration. Series of physic-chemical methods, namely: the compounding (mechanical mixing) method, plasticization, and impregnation of organic and inorganic fillers have been considered in the present study. The tests were conducted using a dynamic mechanical analyzer Q-800 in order to determine the dynamic characteristics of the materials as a function of time, temperature, and frequency following double cantilever geometry. Modifiers that provide the best mechanical properties, including epoxy silicate iron, graphite, and calcium stearate, have been defined by the results of static and dynamic mechanical tests. As follows from the plots presented in Fig. 8 dependence of the dynamic modulus of elasticity on the concentration of montmorillonite (MMRT) nanofiller (with varying from 1 to 10%) has an extreme kind. The maximum value of Ed is reached at 5 mass% of montmorillonite concentration.

The dependence of the dynamic modulus of elasticity on the mass concentration of montmorillonite correlates with the results of acoustic tests of sandwich structures with a damping layer of the composites based on epoxy resin, as presented in Fig. 9. It is very important to note that introduction the montmorillonite nanofiller of optimum concentration in epoxy matrix provides increase in the sound-transmission in the low and medium frequency ranges up to 40 dB, and has no significant effect on the density of the composite material for damping layer. As a result, it does not increase the total weight of the layered composite structure.

New formulations of epoxy composites containing nano-sized silicate fillermontmorillonite, providing an increase in the coefficient of mechanical losses of up



Fig. 8 Temperature dependences of the storage modulus and mechanical loss tangent of epoxy composites of different compositions: *1* Initial composition (epoxy resin without fillers); 2 Composition with 1 wt% MMRT; *3* Composition with 5 wt% MMRT; *4* Composition with 10 wt % MMRT



Fig. 9 Transmission loss of the materials and layered structures: I Foamed polyurethane (thickness of 25 mm); 2 Steel plate (thickness of 1 mm); 3 Absorbing and damping layers without a steel plate (thickness of 8 mm); 4, 5, and 6 Structures with a damping layer based on nano-modified epoxy resin containing 10, 1, and 5 wt% MMRT, respectively

Tape common view		
Technical drawings		N N N N N N N N N N N N N N N N N N N
Туре	Α	С
Steel	08ps-OM-T-2-T	50-T-S-N
Standard	GOST 503-81	GOST 2284-79
$F_{t,\max}$ (N)	5539.86	10052.61
$\sigma_v (\text{N/mm}^{2)}$	220.65	406.81
$\Delta l_{t.pl.}$ (mm)	6.54	2.25
Relative strain $\varepsilon_{F,\max}$	0.039320	0.012107

Table 5 Metal punch tapes used samples main characteristics

to 30%, offset $tg\delta_{max}$ at lower temperatures on 25–30 °C and rising to 20% of $tg\delta_{max}$ were proposed in result of studies. Wherein the incorporation basalt fibers into the composite as reinforcing fillers compensates undesirable reduction of dynamic modulus associated with the introduction of the montmorillonite and increases it by 8.3% compared to the initial composition.

3.3 Using of Perforated Metal Tapes

Utilization of two types of perforated metal tapes in polymer resins has been investigated for an application in pipelines repair. Main characteristics of metal punch tapes are present in Table 5. The mechanical properties of obtained composites (Fig. 10a) have been evaluated by three-point bending tests (Fig. 10b). Results are present in Fig. 10c. Application of perforated metal tape in polymer resin composites demonstrates increasing of mechanical strength, which is very important for material used for repairing of pipelines.



Fig. 10 Polymer-metal punch tape composite sample in translucent lighting (a), three-point deformation test (b), and their test results (c)

4 Conclusions

- (i) We proposed original technique for determining the mechanical properties of a composite material based on glass fibers and epoxy resin.
- (ii) Introduction of disperse fillers in epoxy resin had a positive effect on the mechanical properties of examined composites.
- (iii) Concentration of montmorillonite nanofiller had a significant impact on dynamic modulus of elasticity of composite materials based on epoxide resin.
- (iv) Application of perforated metal tape in polymer resin composites demonstrated increasing of composite mechanical strength.

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