Experimental determination of the adhesive characteristics of the “elementary fiber-epoxy matrix” system

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Abstract. The paper considers the method of testing the elementary fiber-polymer matrix system. Results of determining the following adhesive characteristics are provided: apparent and local adhesive strength, start and end in the adhesive destruction of the fiber-matrix joint under study. Basalt fiber and epoxy matrix based on the epoxy resin and amine hardener are considered as the study objects. Influence of the samples geometric characteristics on their adhesive characteristics is established. The paper shows that at the depth of the elementary fiber emersion in the polymer matrix equal to 200 μm, the scatter of all determined adhesive characteristics is significantly reduced.

1 Introduction

Basalt plastics belong to the group of reinforced polymer composite materials (PCM), which operation scope is constantly increasing [1-4]. Basalt fibers are comparable to the glass fibers according to their deformation-strength and thermophysical characteristics; however, if the properties of PCM based on the glass reinforcing fillers are well studied [5-12], technical literature contains only a very limited number of works devoted to studying basalt fibers and composites on their basis.

The authors of works [1-3, 13] show the prospects of using vacuum infusion technology in the PCM production based on the basalt fibers. This molding technology is widely used in manufacturing parts made of fiberglass, because it allows significantly reducing the cost of finished parts made of the composite materials.

Many standard methods are used to evaluate the PCM mechanical characteristics making it possible to determine their values under static and dynamic conditions at tension, compression, bending, etc. Researchers pay much less attention to experimental evaluation of the adhesive strength, although it is known [4, 14] that exactly the composite structure durability depends on it.

The authors of [14, 15] are using the pull-out method to evaluate adhesive strength in the polymer matrix–fiber system. Essence of the method is to experimentally determine the force, at which the elementary fiber is being pulled out (without destruction) of the cured

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matrix. Based on the results of these tests, a certain average value of the adhesive strength is determined, but the influence of residual stresses, friction forces and other factors that have a significant effect on the adhesive characteristics is not taken into account.

Objective of this work is a comprehensive evaluation of the adhesive strength characteristics of the elementary basalt fiber–epoxy matrix system.

2 Research objects

Research objects included elementary basalt fibers (Table 1) and epoxy binder (Table 2).

Table 1. Elementary basalt fiber characteristics

Characteristics	**Indicators**
Fiber mark	KamennyVekNRB-14-400-KV Direct
Diameter, mcm	13−15
Strength limit at extension, MPa	2900−3200
Elasticity module, GPA	85−90

Table 2. Epoxy binder and cured matrix characteristics

Characteristics	Values
Binder characteristics	
Number of components	2
Resin – curing agent ration	10:1
Curing mode: Temperature, °C Time, min	 60 20
Viscosity, Pa·s, at the shift rate of 67 1/s	5.8
Cured matrix characteristics	
Strength limit at bending, MPa	105
Strength limit at compression, MPa	62
Strength limit at extension, MPa	50
Vitrification temperature, °C	58

Polyethylenepolyamine was used as the curing agent, and the ED-20 epoxy oligomer - as the resin. This material cures at the room temperature for 24 hours, and it was cured at the high temperature in order to reduce duration of the sample preparation.

3 Methods for experimental evaluation of the adhesive characteristics of the elementary fiber-epoxy matrix system

Samples were prepared and tested using the Textechno equipment consisted of two modules: Favimat+ and Fimabond (Table 3) [15].

Table 3. Characteristics of the Textechno equipment used fin manufacture and pull-out testing

Indicators	Values
Fimabond	
Purpose	Sample manufacture

Internal diameter of a cup, where the binding agent was flooded, mm	2
Elementary fiber emersion depth into the binding agent, mcm	100 – 300
Emersion depth accuracy, mcm	10
Elementary fiber diameter accuracy, mcm	0.01
Elementary fiber emersion in the binding agent rate, mcm/min	100 – 150
Favimat+	
Purpose	Pull-out method testing
Destruction load accuracy, cN	0.02
Rate, mm/min	0.1

Technique used in manufacturing the samples for their pull-out testing consisted of the following main operations:

- binder manufacture, pouring it into the cup and installing the cup with the binder into the Fimabond module;
- elementary fiber extension from the multifilament thread, which was used to produce the fabric, its capture in the device, installation of the device with elementary fiber in the Fimabond module, fiber immersion in the binding agent and curing the binding agent.

The finished sample was put into the Favimat+ module, where a load was applied to the fiber, and the force was determined at the room temperature, at which the elementary fiber was extracted (without destruction) from the cured polymer.

As a result of experimental research, dependence of force on displacement was obtained, it is shown in Fig. 1.

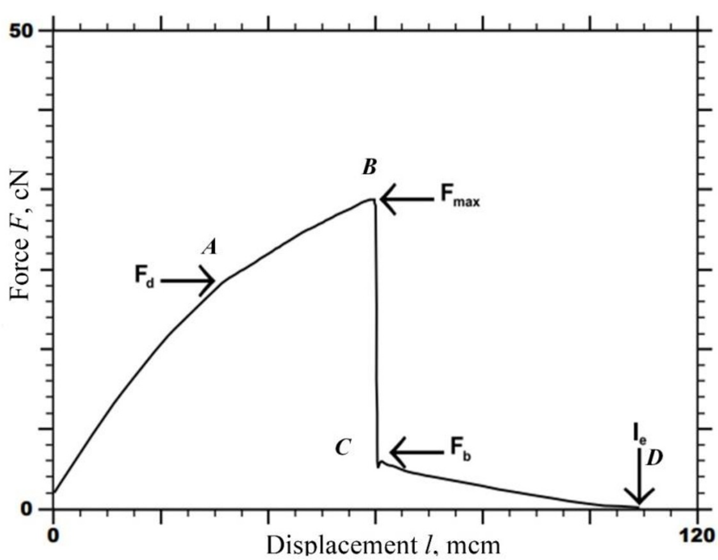

Fig. 1. Dependence of force on displacement during testing the elementary fiber-polymer matrix system

The following notations are used in Fig. 1:

- F_d is the force applied to the adhesive joint initiating the crack growth and leading in turn to the adhesive failure.

- F_{max} is the maximum force, at which the adhesive joint is being completely destroyed ($F_d = F_{max}$ value in Table 4).
- F_b is the minimum frictional force between the polymer matrix and the fiber appearing during destruction of the adhesive joint.

As the adhesive connection initial characteristics, the following notations are used:
- d_f - individual fiber diameters;
- l_e - depth of elementary fiber immersion in the polymer matrix [mcm].

4 Methods for theoretical evaluation of the adhesive characteristics of the elementary fiber-epoxy matrix system

Based on the results of experimental studies, for each adhesive joint and in accordance with the procedure [16, 17], the following is determined:
- W_{de} adhesive destruction operation, which is assessed by the area under the curve (Fig.) in the OB section.

$$W_{de} = \int_0^{l_{Fmax}} F \cdot dl \tag{1}$$

- W_{fric} work spent on overcoming the friction forces, which is determined by the area under the curve (Fig.) in the CD section.

$$W_{fric} = \int_{l_{Fmax}}^{l_e} F \cdot dl \tag{2}$$

- τ_{app} apparent adhesive strength (MPa), which is characterized by the average shear stress at the interface at the maximum load.

$$\tau_{app} = \frac{F_{max}}{\pi \cdot d_f \cdot l_e}, \tag{3}$$

- τ_f interfacial friction stress (MPa), which characterizes the stress resulting from the fiber surface friction against the matrix and corresponding to the CD section on the force-displacement curve.

$$\tau_f = \frac{F_b}{\pi \cdot d_f \cdot l_e} \tag{4}$$

- τ_d local adhesive strength (MPa), which represents the shear stress average value at the matrix-fiber interface under a load corresponding to the adhesive joint complete destruction.

$$\tau_d = \frac{F_d \cdot \beta}{2 \cdot \pi \cdot r_f} \cdot \cot(\beta \cdot l_e) + \tau_T \cdot \tan\left(\frac{\beta \cdot l_e}{2}\right), \tag{5}$$

where: τ_T are the residual thermal stresses; r_f is the fiber radius; β is the coefficient, which the authors of this works [16, 17] called the shift delay parameter.

$$\beta = \left[\frac{2}{r_f^2 E_f E_m}\left[\frac{E_f v_f + E_m v_m}{\frac{v_m}{4G_f} + \frac{1}{2G_m}\left(\frac{1}{v_m}\ln\frac{1}{v_f} - 1 - \frac{v_f}{2}\right)}\right]\right]^{1/2},$$

where: E_m, E_f are the matrix and the fiber Young moduli; G_f is the fiber shear modulus; v_f and v_m are volumetric fractions of the fiber (f) and matrix (m), respectively.

5 Results and discussion

Table 4 presents the results obtained in determining adhesive characteristics for two types of samples, which differed from each other in immersion depth of the elementary fiber in the polymer matrix.

Table 4. Results of establishing adhesion characteristics in the basalt fiber – epoxide matrix system

No.	F_d	F_b	l_e	τ_{app}	τ_d	τ_f	W_{de}	W_{fric}	DF_{max}	F_{max}	d_f
	cN	cN	mcm	kPa	kPa	kPa	J	J	mcm	cN	mcm
Fiber immersion depth in matrix of 200 mcm											
1	26,2	8,9	197,1	36457,9	72302,6	8999,2	8,9	4,8	38,2	36,1	16
2	24,1	8,7	189,8	35852,9	67323,6	9121,3	9,3	4,2	42,2	34,2	16
3	26,5	9,4	213,8	32091,4	72571,4	8732,7	8,5	5,3	47,6	34,5	16
4	23,2	4,8	219,1	32781,8	64565,2	13458,3	7,4	9,9	34,3	36,1	16
5	23,3	7,7	193,2	30893,3	65242,4	7917,9	6,2	3,7	40,0	30	16
Fiber immersion depth in matrix of 100 mcm											
6	16,1	7,0	108,1	39639,7	52209,2	12899,1	2,3	2,3	17,9	21,5	16
7	15,91	4,59	99,35	36628,8	53127,4	9199,2	2,2	1,6	18,	18,3	16
8	12,25	4,02	84,7	36085,0	44728,4	9438,6	1,3	1,3	13,9	15,4	16
9	10,63	4,97	76,7	42811,8	73610,7	12879,1	1,7	1,0	24,6	16,5	16
10	10,43	5,61	117,9	30552,9	80610,7	9471,7	2,1	1,5	26,3	18,1	16

Results of the research demonstrated that the elementary fiber immersion depth in the matrix equal to ≈100 μm was not sufficient, because of a large data scatter. Thus, the τ_d local adhesive strength values were varying from 44,728.4 kPa to 80,610.7 kPa, i.e. by 1.8 times. It should be noted that for the same materials at the immersion depth equal to ≈200 μm, error in the local adhesion strength was not exceeding 12% (the τ_d local adhesion strength values varied from 64,565.2 kPa to 72,571.4 kPa).

It was established that for all the studied samples, the apparent adhesive strength value was significantly higher than the friction stresses arising after the adhesive joint destruction due to fiber extension from the polymer matrix. The local adhesive strength value, also for all the samples under study, was higher than the apparent adhesive strength value. Thus, the local adhesive strength value actually reflected the true adhesive strength value of the elementary fiber-polymer matrix system.

For specimens No. 6 and No. 8 (see Table 4), the value of work spent on the adhesive joint destruction was equal to the work of the friction forces occurring already after the adhesive joint destruction. For the sample No. 4, its value even exceeded the work expended on destructing the adhesive joint. That was probably due to high roughness of the samples No. 4, No. 6 and No. 8. However, higher roughness values of those samples were not leading to a similar increase in the local adhesive strength values.

6 Conclusion

The pull-out method used in this work makes it possible to determine the adhesive strength values with a high degree of accuracy.

As the research results, the following adhesive characteristic values were experimentally determined: work that should be performed for adhesive destruction, work associated with overcoming the friction forces after the adhesive destruction, as well the apparent and local values of the adhesive strength. It was established that the work value that should be performed on the adhesive joint destruction could be equal to similar work occurring after destruction of the matrix-fiber interface.

It was also found that in order to reduce the spread in all the determined values of the adhesive characteristics, it was necessary that the fiber immersion depth in the matrix should be ≈200 μm.

The proposed method could be widely used in design and development of new coupling agents and technologies for their application to fiber, since it would allow evaluating the coupling agent material contribution to the adhesive strength value in the elementary fiber-polymer matrix system.

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