INNOPIPES
INNOVATIVE NONDESTRUCTIVE TESTING AND ADVANCED COMPOSITE REPAIR OF PIPELINES WITH VOLUMETRIC SURFACE DEFECTS

European Commission

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Paton Welding Institute
Kiev, Ukraine

Protocol of composite material and its components test

Contents

Roman Dmytriienko dri1@ukr.net, www.dri1.cc.ua April 2015

1. The material of the reinforcing element and the resin

As the reinforcing element is used straight roving (fig. 1) from fiberglass EC 10 1680H-У10(168) [2], as the filler - epoxy resin КДА-ХИ [1]. Here and further in the text of the Protocol, the roving – is the single strand of fiberglass, consisting of ~ 1200 filaments.

Table 1. The results of roving EC 10 1680H testings[1)]

№	Parameter	EC 10 1680H-У10(168)[3)]	EC 10 1680H-У11(168)
1	Linear density, teks	1760	1748
2	Moisture content,%	0.02	0.02
3	Substances deleted during roasting,%	1.0	0.81
4	Unit breaking load, mN/teks[2)]	599.1	653.4

Notes:
[1)] Tested on 28.04.2005 by " Объединение стеклопластик " Ltd according to [4, 5]. For winding was used the roving EC 10 1680H-У10(168) with the lubricant У10, the results of the roving in combination with lubricant У11 are given for reference;
[2)] 1teks = 1 g/1 km;
[3)] Designation of the roving according to [2]: E - general purpose glass; C - continuous thread; 10 - nominal diameter of elementary filament, μm; 1680 - nominal linear density, teks; H - for winding; У10 - lubricant; 168 - linear density of complex threads, teks.

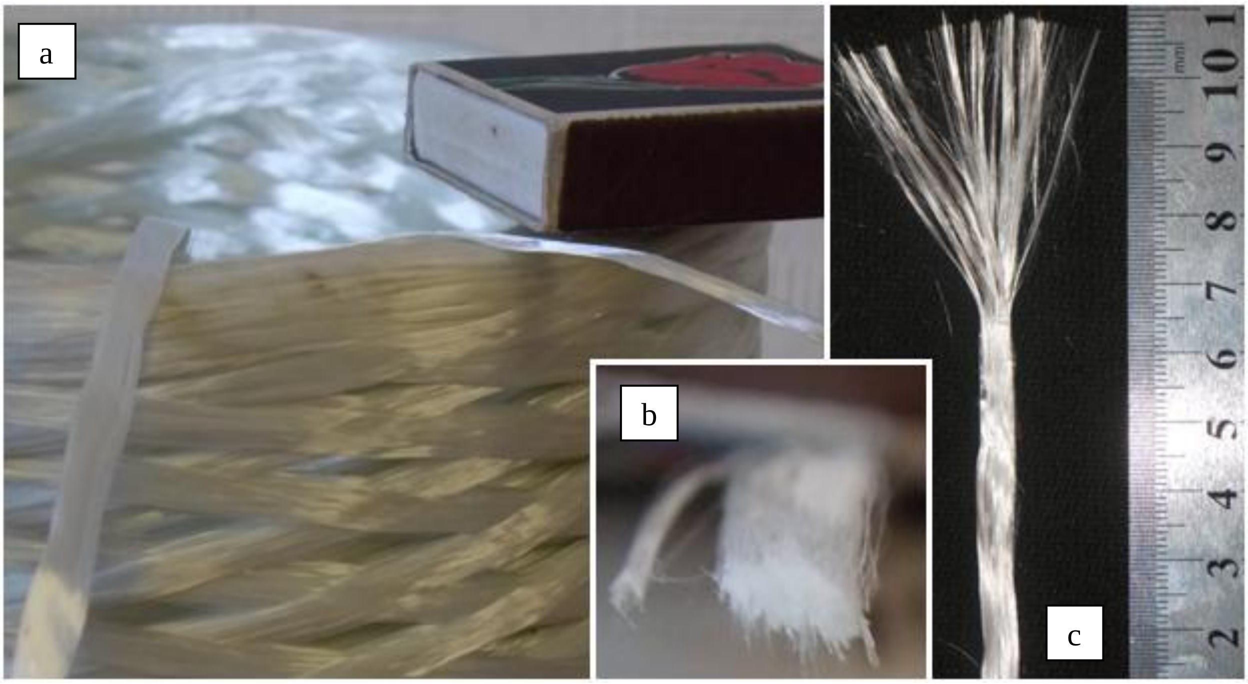

Figure. 1. The roving used as the reinforcing element: a - roving, laid in the reel; b - cross section of roving; c - the end of the roving in free state.

According to [2] minimal unit breaking load of the roving: 396 mN/teks (40 g/teks).

Theoretical breaking load is calculated as the product of the linear density and the unit breaking load:
У10: 1760 × 599.1 = 1054416 mN = 1054.416 N = 107.5205 kgf.
У10: 1748 × 653.4 = 1143891 mN = 1143.891 N = 116.6444 kgf.

Table 2. Characteristics of epoxy resin КДА-ХИ according to the certificate[1)]

№	Characteristic	According to [1]	Actually
1	Appearance	Homogeneous liquid of medium viscosity, from light yellow to light brown color without mechanical inclusions and clots	
2	Viscosity by viscometer VZ-246 (6 mm nozzle diameter) at 25 ± 2 °C, min	0.5-2.0	0.1
3	Gel time (sec) of 2 grams at temperature: 180 ± 2 °C 160 ± 2 °C	50-105 80-120	94

Note:
[1)] Date of manufacture 7.04.2005, warranty period - 12 months.

Manufacturer's information (NPP «Synthesis», Donetsk) on the strength characteristics of the polymer matrix of the resin КДА-ХИ after the standard mode curing (100…180 °C during 2 hours):

-flexural strength - 90...110 MPa;
-tensile strength - 75...80 MPa;
-impact strength - 5...10 Kj/mm^2;
-elongation - 5.5...8%.

Additional information.

- after winding on a cylindrical pipe the roving cross-sectional dimensions typically are 3×0.3 mm;
- after winding on tube with a diameter of 320 mm the density of filling was 0.7...0.72;
- the tension of the roving with force > 5 kg results in breaking of some filaments;
- filling density is defined as the ratio of the weight of glass it has after thermal removal of the resin at a temperature of 400…450 °C during 2…3 h, to the initial weight of the material;
- polymerization of composite repair after its installing is done at a temperature of 120…180°C. The same temperatures was used for polymerization of composite test specimens.

2. Preparation of the tensile test specimens of composite material

To determine the mechanical properties of the composite material specimens in form loop were manufactured. Specimen consisted of two branches, a and b (fig. 2, 3). The loop formed by winding the roving, soaked with epoxy, on special rig (fig. 2a, 2e). During the winding the roving was stretched by applied force. (table 3).

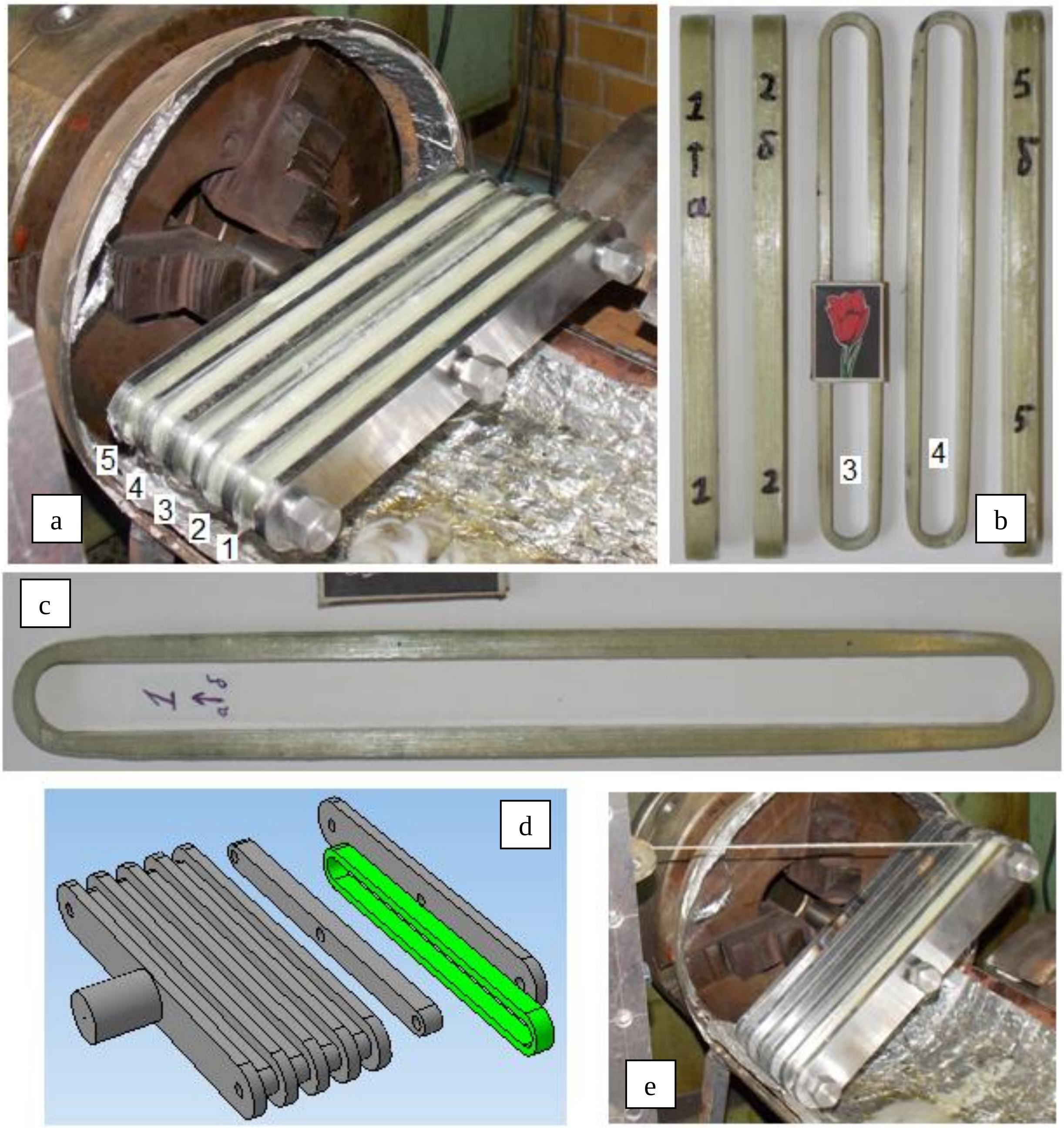

Figure. 2. Preparation of the tensile specimens: a - loops 1... 5, wrapped up in a snap; b - specimens of П1…П5 before the test; c - sample П1; d - model of used rig; e - winding loops on the rig.

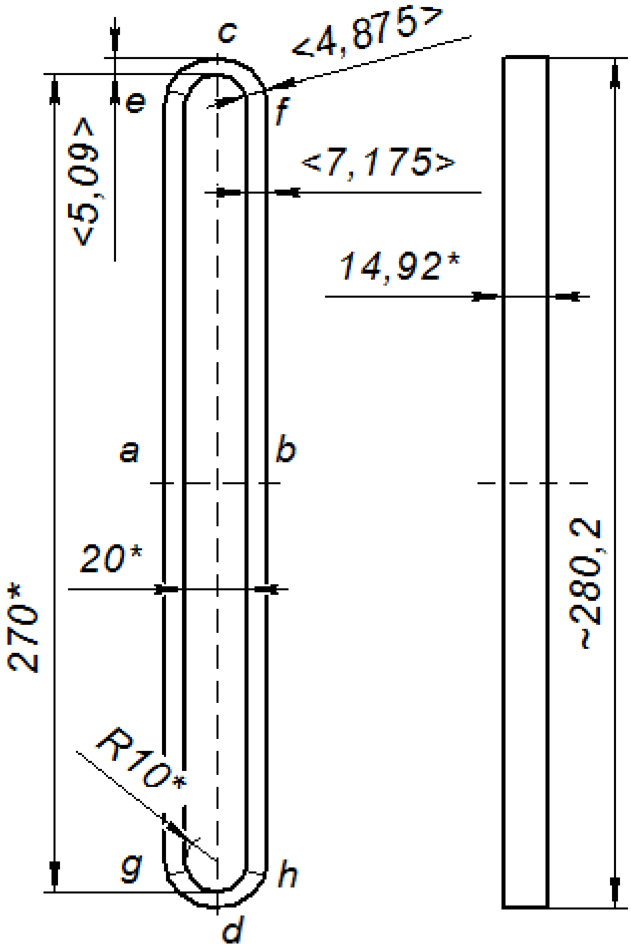

Figure. 3. The geometrical dimensions of the tensile specimen: * - dimensions are the same for all specimens; < > average size; (a), (b), (c), (d), (e), (f), (g), (h) - sections of specimen.

Table 3. Characteristics of tensile specimens.

Specimen			П1	П2	П3	П4	П5	**Average**
[1] Preliminary tension of roving, N_1, *kg*			2	4	3	3.5	0.1	**2.5**
[2] number of turns of the roving			80	80	80	80	75	**79**
width of the section, mm	(a)		7.3	6.8	6.7	7.0	8.0	**7.2**
	(b)		7.1	6.8	6.5	7.0	8.6	
	(c)		4.9	5.1	5.3	4.9	5.2	**5.1**
	(d)		4.9	5.0	4.9	5.2	5.5	
	round place	(e)	4.8	4.6	4.8	5.0	5.0	**4.9**
		(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	
Fracture location			(g)	(f)	(e)	(e)	(g)	

Notes:
[1] average value, amplitude variation during winding ± 1.5 kg;
[2] number of turns may vary ± 3.

3. The test results of the composite material

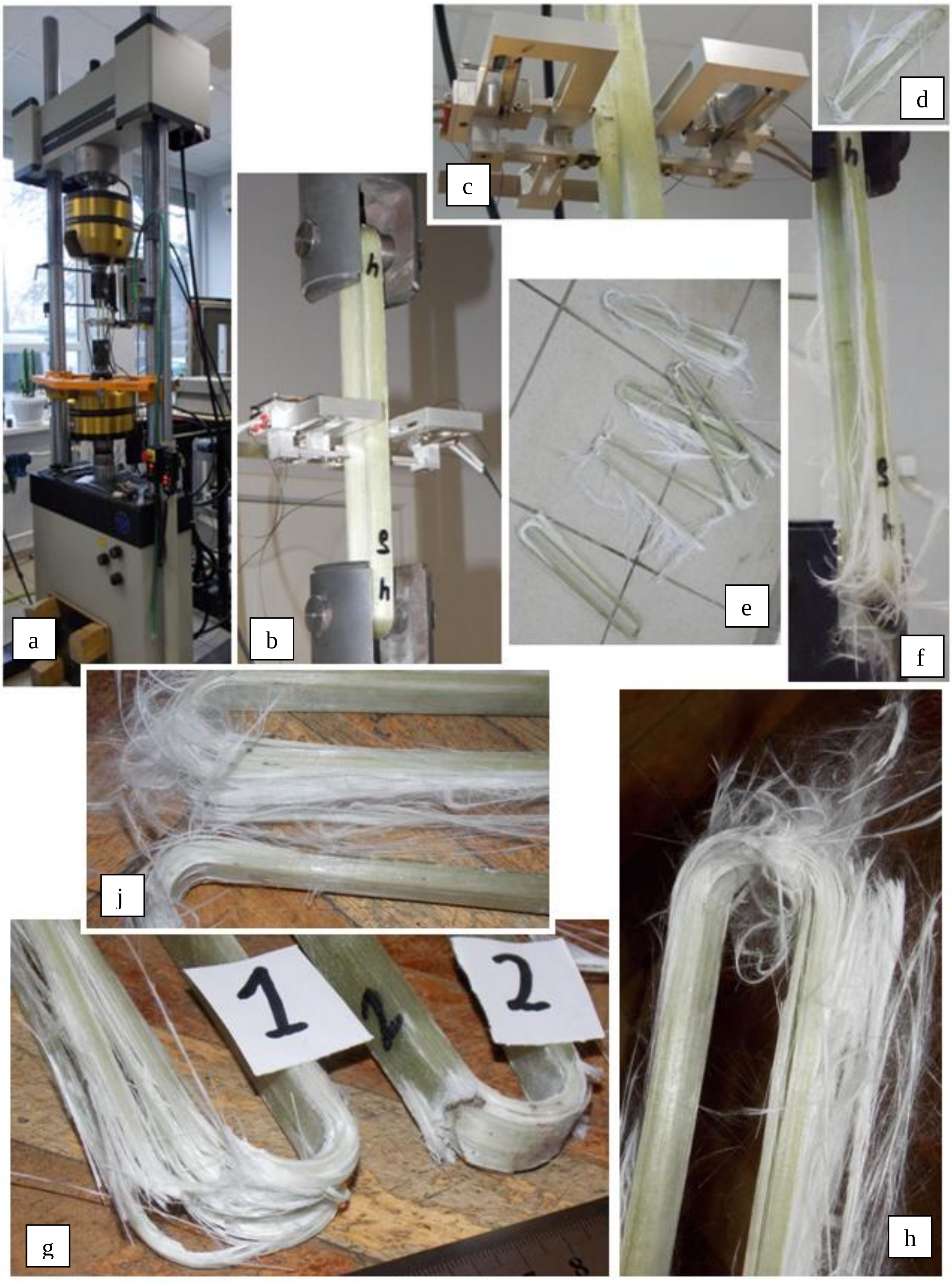

Figure. 4. Tensile test: a - testing machine Instron 8802; b - specimen gripping; c - extensometer Biaxial 2620-614; d, e, f, j, g, h - features of specimens fracture.

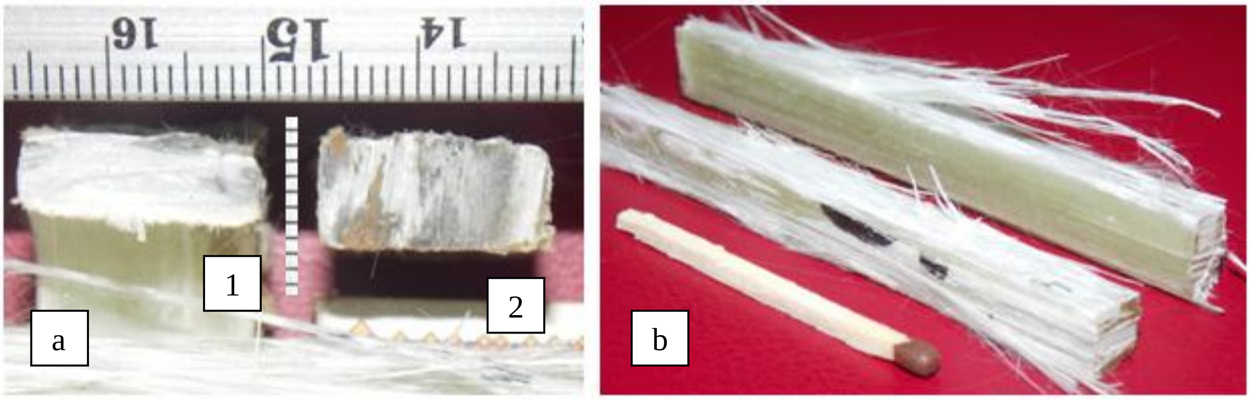

Figure. 5. Specimen Π4 after the destruction: a - cross section (1 – at the section *c*, 2 - at the section *a*, (see Fig. 3); b - longitudinal splitting of branche *a* (see Fig. 3).

Table 4. Results of tensile tests[1)].

Specimen	Π1	Π2	Π3	Π4[3)]	Π5	**Average**
stress of fracture, kN	95.28	101.23	99.19	96.04	99.87	**98.32**
max. strain,%	1.36	1.44	1.59	1.39	1.58	**1.47**
Poisson's ratio μ	0.179	0.196	0.164	-	0,147	**0.172**
[2)] Young's modulus, E, MPa	32721	34645	31673	33079	25518	**31527**
[2)] tensile strength, MPa	445.0	498.9	503.6	459.8	403.2	**462.1**

Notes:
[1)] Longitudinal and transverse base of was - 25 mm and 15 mm, respectively. Test temperature + 20 ^{o}C , speed of deformation 10 mm/min.
[2)] The thickness in the middle part of the loop was used in the calculation of the mechanical properties of composite;
[3)] During the test, the shift of the extensometer had occurred.

In Figure 6 are given examples of the specimen Π2 tension.

From fig. 6d we can see that stress F_1, per one roving, almost in proportion to the longitudinal deformation, so after removing of the extensometer the destruction deformation can be found by linear continuation of relation up to ultimate load on one roving.

Summary of the test results of specimens Π1…Π5, given to the force on 1 roving, are presented in Figure 7.

It should be noted that dependence of the destruction and ultimate deformation on preloading of the roving was not detected (fig. 8).

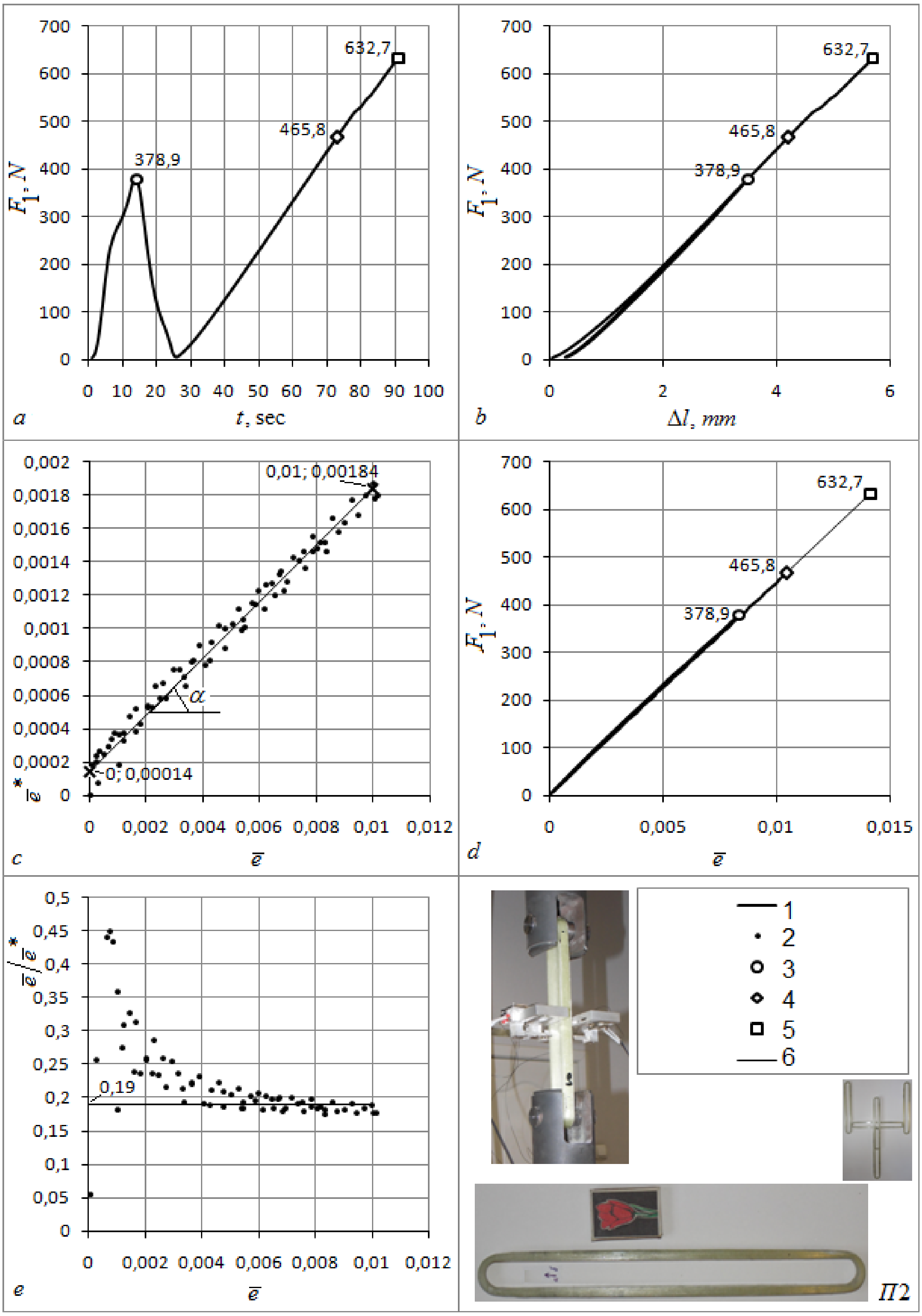

Figure. 6. Tension of the specimen П2: a, b, d - dependence of force per 1 roving (F_1) from time (t), displacement of grips (Δl) and strain ($\bar{e}$), respectively; c, e - transverse strain ($\bar{e}^*$) and ratio ($\bar{e}/\bar{e}^*$) depending on the longitudinal deformation ($\bar{e}$). 1, 2 - test results; 3 - load-shedding; 4 - removal of extensometer; 5 - ultimate load; 6 - linear relationship.

Table 5. Longitudinal deformation $\bar{e}$ depending on the force per 1 roving F_1 and on the parameter $E_1 = \frac{F_1}{\bar{e}}$.

Specimen	Load shedding			Removal of extensometer			Fracture	
	$\bar{e}$	F_1, N	E_1, N	$\bar{e}$	F_1, N	E_1, N	$\bar{e}_{max}$ calc.	F_{1max}, N
П1	0.008453	380.8705	45057.44	0.010409	465.1008	44681.88	0.013328	595.5324
П2	0.008336	378.9419	45458.48	0.010417	465.8148	44714.94	0.01415	632.7281
П3	0.00835	357.5832	42824.34	0.010496	450.6579	42937.59	0.014439	619.9876
П4	0.008488	427.4154	50355.25	0.011518	556.4347	48308.12	0.012426	600.2789
П5	0.010759	449.5257	41781.36	0.015899	665.8315	41877.96	0.015899	665.8315
Average	**0.008877**	**398.8673**	**45095.37**	**0.011748**	**520.7679**	**44504.1**	**0.014049**	**622.8717**

Notes: F_{1max} - fracture force per 1 roving; $\bar{e}_{max}$ calc. - design value of ultimate deformation; the value of E_1 is the same as at the moment of the extensometer removing.

Table 6. Some clarifications of the tensile test.

Specimen	Fracture force, N	Ratio $\lvert \bar{e}_* / \bar{e} \rvert$ For $\bar{e}$>>0 (see fig. 6e)	The tangent of an angle α (see fig. 6c)
П1	95285.18	0.18	0.18
П2	101236.5	0.19	0.17
П3	99198.01	0.17	0.17
П4	96044.63	-	-
П5	99874.73	0.15	0.15
average	**98327.81**	**0.173**	**0.168**

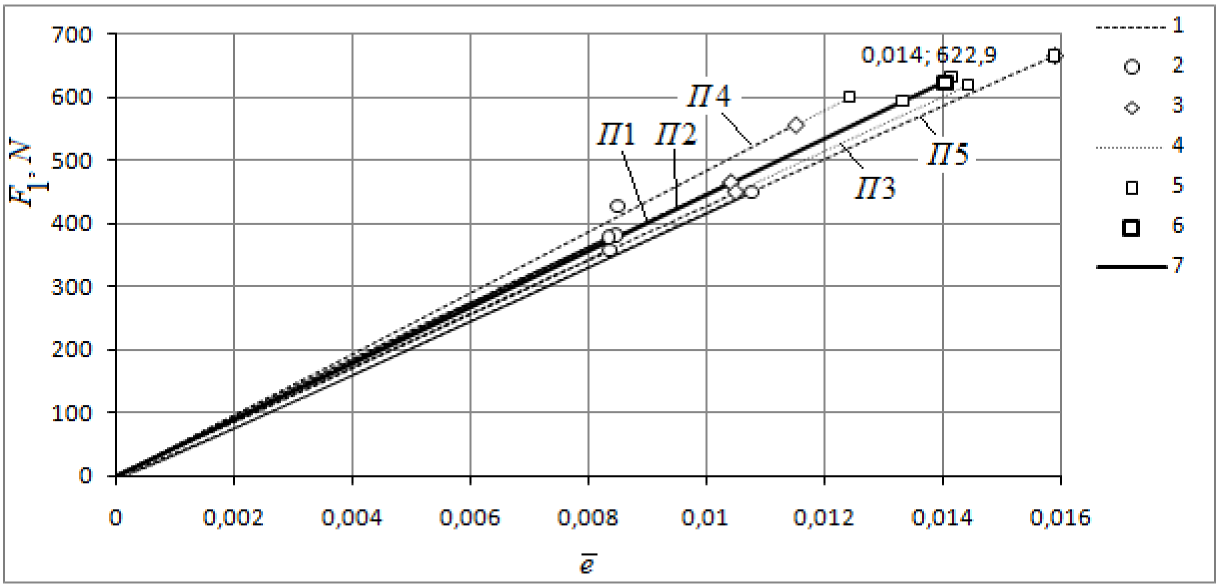

Figure 7. Tension of 1 roving (samples of П1…П5): 1 - according to extensometer; 2 - load shedding; 3 - removal of extensometer; 4 - linear continuation from the condition of proportionality; 5 - limit values the force F_1 and strain $\bar{e}$; 6 - the average maximum value; 7 - average dependence of force F_1 from strain.

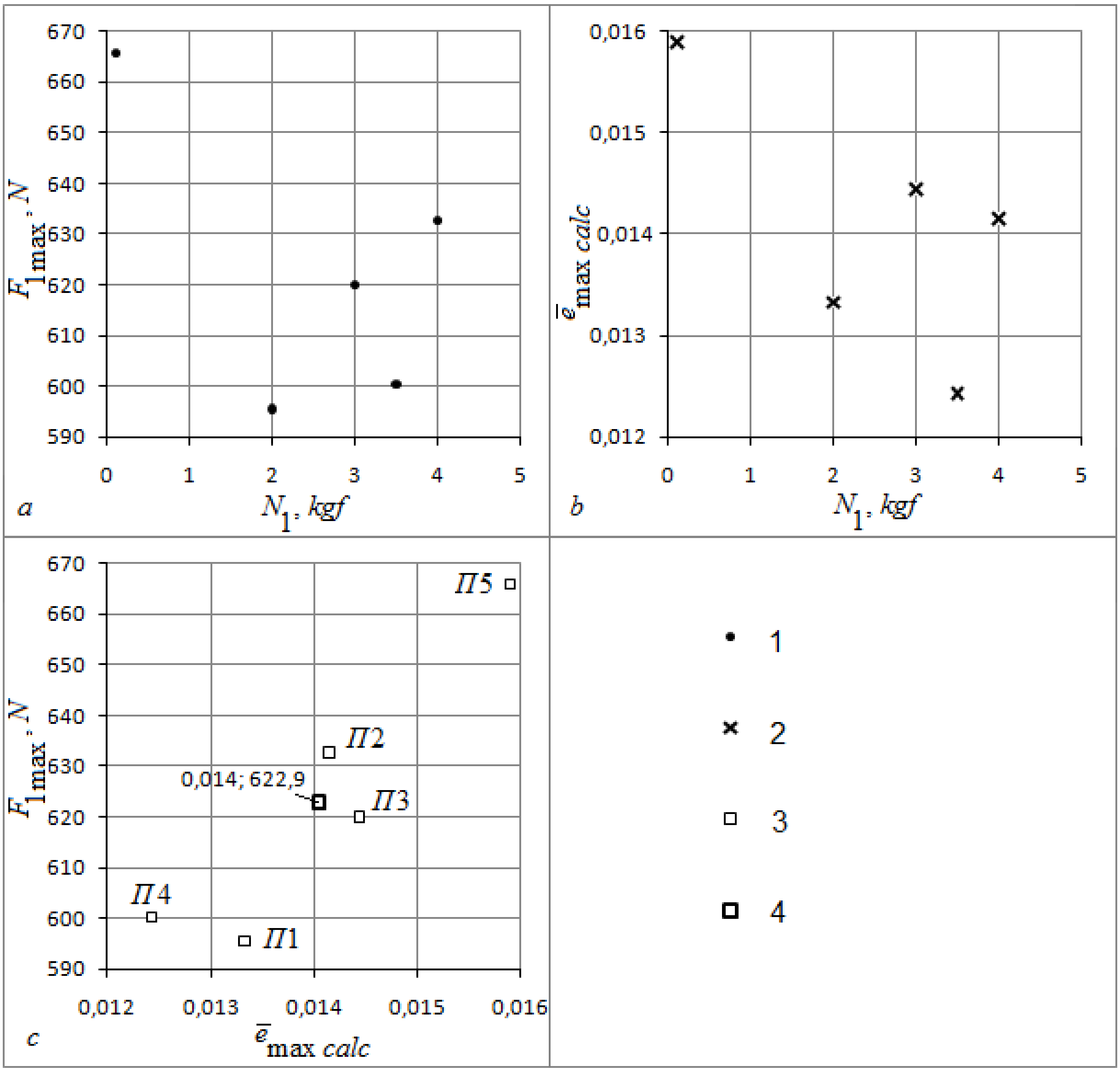

Figure 8. The dependence of the force F_{1max} (applied to 1 roving) - (a) and strain $\bar{e}_{max\ calc}$ - (b) from the tension of the roving N_1 (see Table 3); F_{1max} from $\bar{e}_{max\ calc}$ - (c).

1 - fracture force of the preloaded roving; 2 - ultimate strain of preloaded roving; 3 - the force and strain at fracture; 4 - average maximum values.

4. The tensile test of the roving

In addition to the composite test the roving test was conducted (Fig. 9). The test was carried out on machine Instron 8802 using extensometer 2620-601. Measuring base was 52.5 mm, working section length - 140 mm. Temperature during the test + 20 ^{o}C, speed of displacement - 30 mm/min.

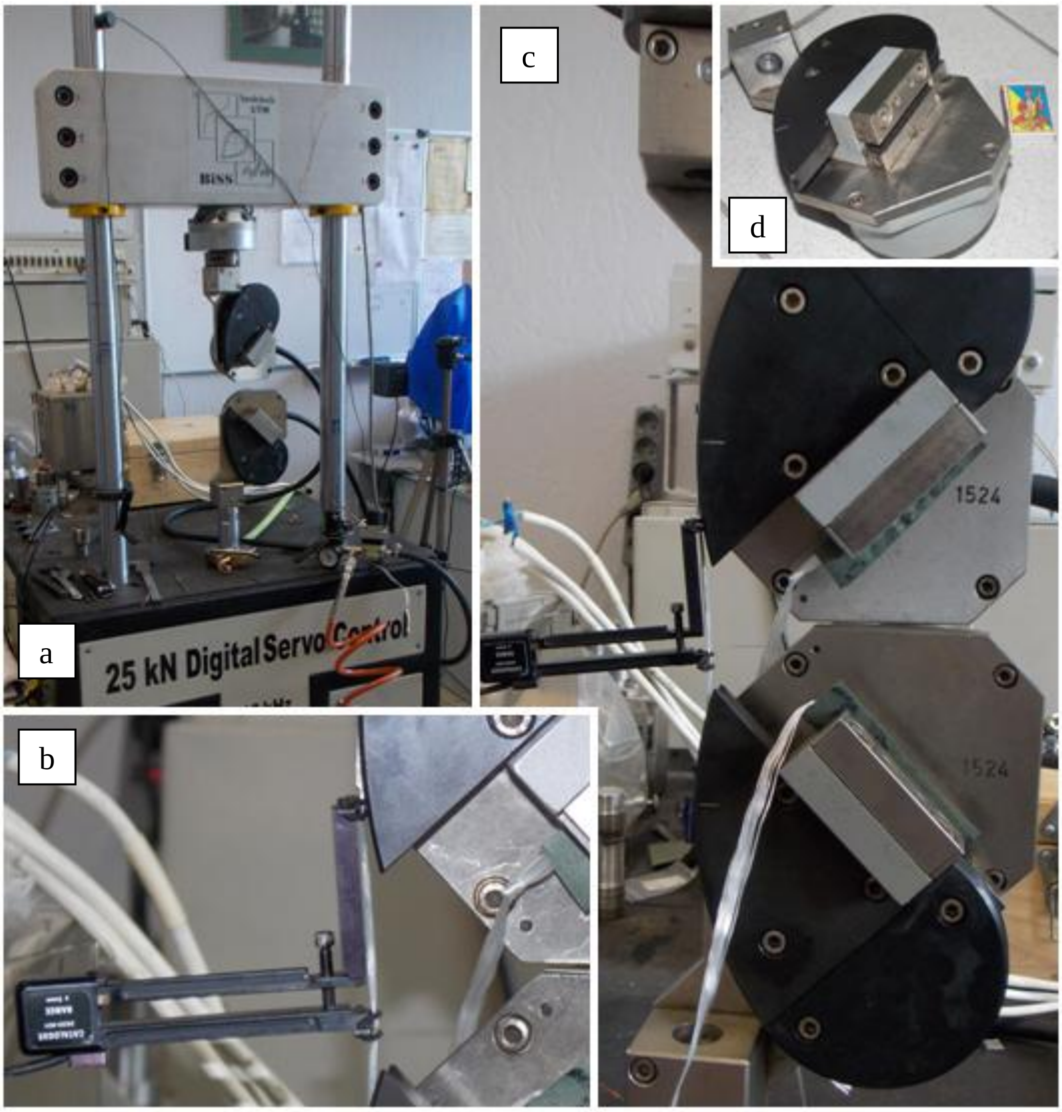

Figure 9. Tensile test of roving: a - testing machine Instron 8802; b - extensometer 2620-601 installed on the specimen; c - general view of the roving test; d - the "snail" grip.

As a specimen was used a section of the roving spooled from reel.

Limit values of strains (Table 7) were obtained by calculation and not by direct measurements. Mechanical properties of the roving were determined on the base of test results of specimens P1, P2, P3 (Table 7). Test results of specimens are presented in Figure 11.

The graphical dependency $\bar{\bar{e}} = f(\Delta l)$ was not available because of instability of extensometer fixation .

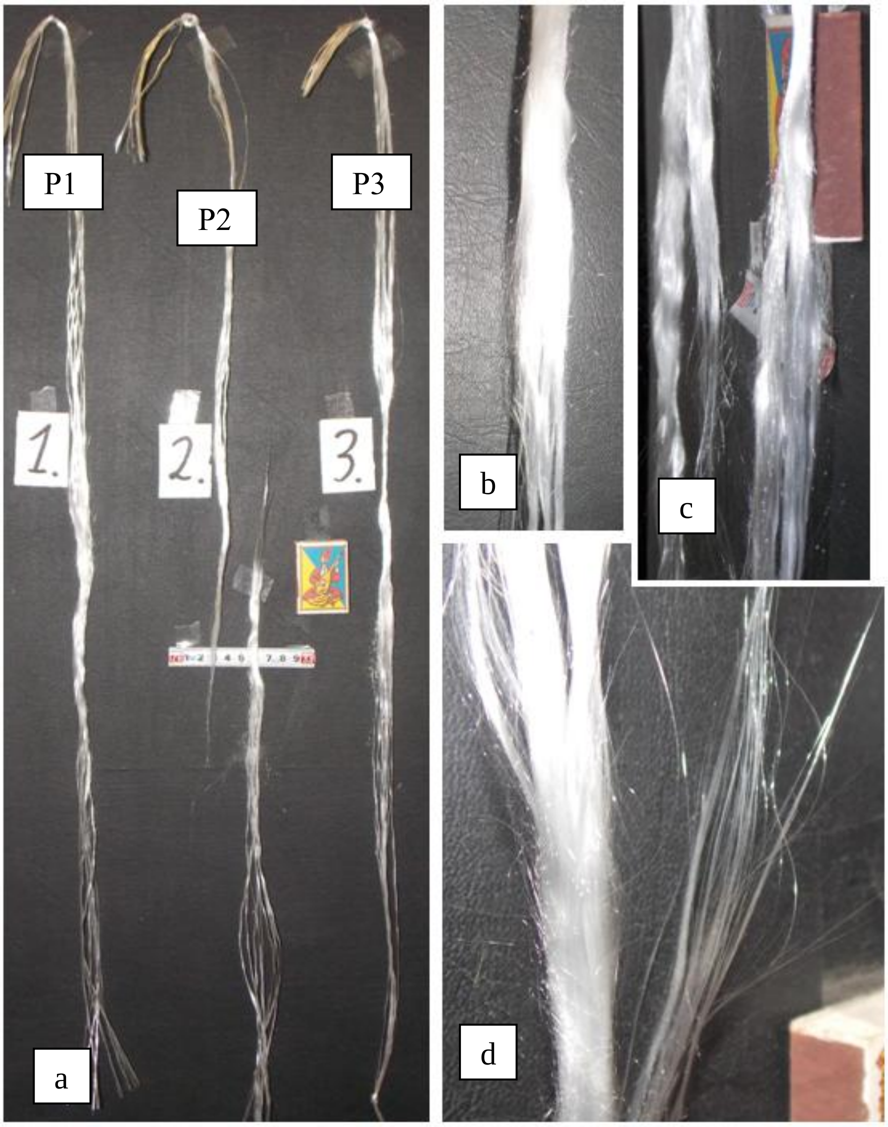

Figure 10. Specimens of roving (P1, P2, P3) after tensile testing.

Table 7. The results of the roving tensile test.

# specimen	fracture force, kN	Max. deformation, %	Rigidity, kN/mm[1)]	Specimen marking	Place of destruction
1[2)]	0.41	1.73	0.91		in grips
2	0.62	1.64	1.55	P1	working part
3	0.64	1.20	0.88		in grips and working part
4	0.55	1.02	1.04		in grips and working part
5[3)]	0.66	-	-		working part
6[3)]	0.73	-	-		working part
7[3)]	0.65	-	-		working part
8	0.69	1.31	1.21	P2	working part
9	0.6	1.70	-	P3	working part

Note:
[1)] Approximate value;
[2)] The sample had the original damage;
[3)] Tests without the use of extensometer.

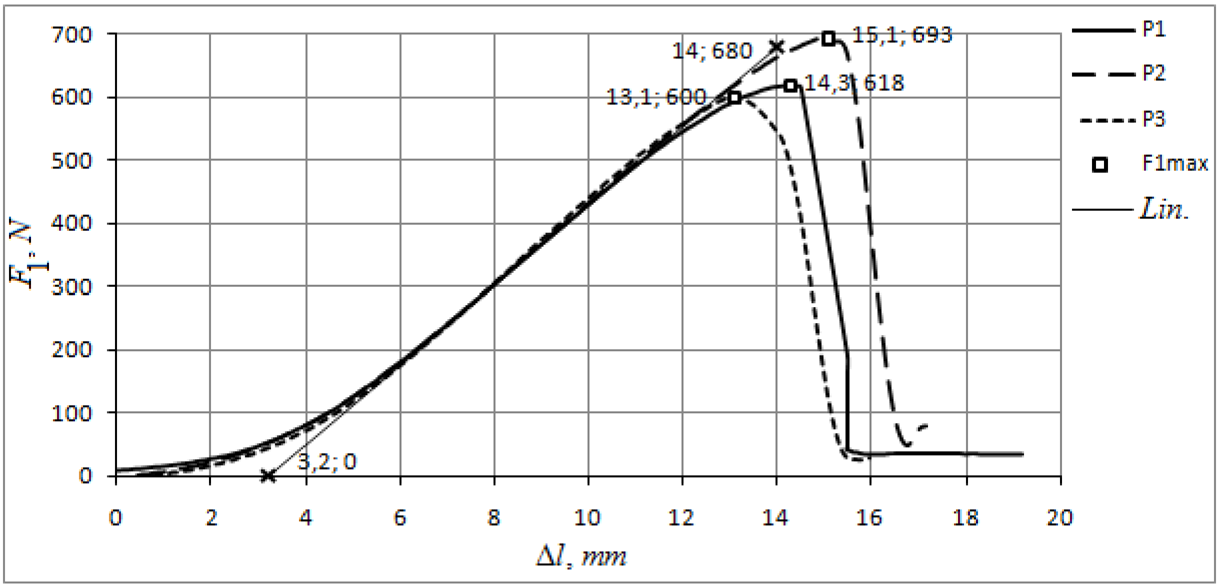

Figure. 11. Dependence of force in specimens P1, P2, P3 from grips displacement Δl. $F_{1\max}$ - maximal force during the test; Lin. - linear dependence.

5. Comparison of test results of the roving and composite material

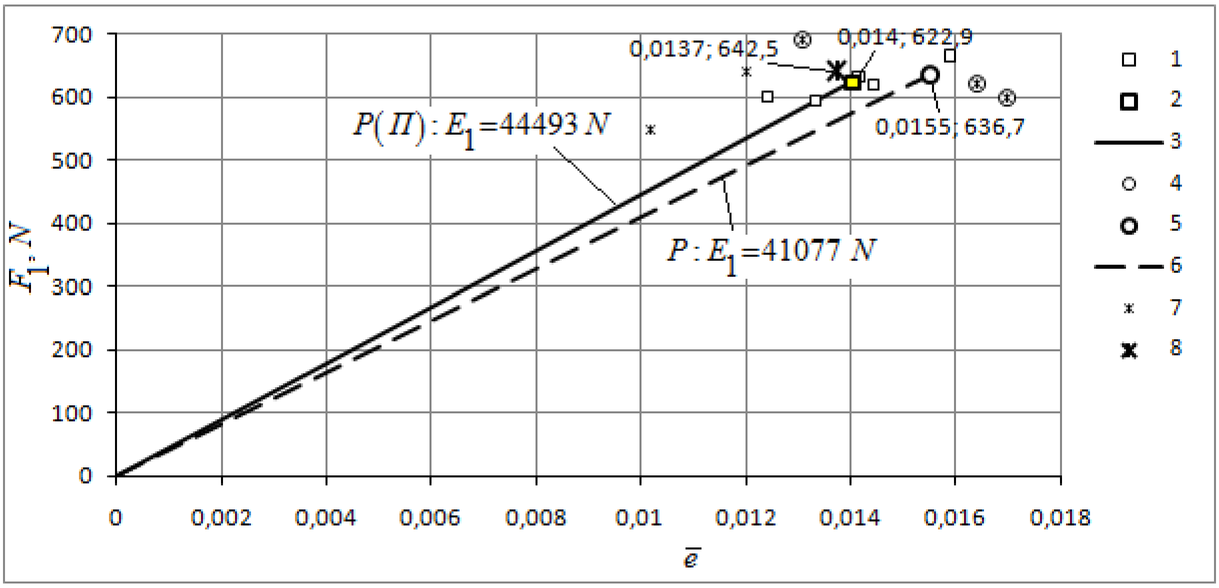

Figure. 12. A summary diagram of tension. Composite test: 1 - limit force F_1 and strain $\bar{e}$; 2 - average maximum value; 3 - average dependence $F_1 = f(\bar{e})$. Roving test (specimens P1, P2, P3): 4 - displacement by extensometer; 5 - load-shedding; 6 - extensometer taking-off; all specimens in Table 7 except specimen 1; 8 - average of all data in Table 7.

Table 8. Test results of the roving specimens and roving as component of П1...П5 specimens.

Roving					Roving of П1...П5 specimens			
Specimen	$\bar{e}$	F_1, H	E_1, H		Specimen	$\bar{e}$	F_1, H	E_1, H
P1	0.0164	620	37805		П1	0.0133	595.5	44682
P2	0.0131	690	52672		П2	0.0142	632.7	44715
P3	0.0170	600	35294		П3	0.0144	620.0	42938
					П4	0.0124	600.3	48308
					П5	0.0159	665.8	41878
Average	**0.0155**	**636.7**	**41924**		**Average**	**0.0140**	**622.9**	**44504**
					min	0.0124	595.5	41878
					max	0.0159	665.8	48308

In calculations could be used average values of specimens П1…П5 test data: the limit strain $\bar{e}=0{,}014$; the limit force per roving $F_1=622{,}9\,H$, and from here, $E_1=F_1/\bar{e}=622{,}9/0{,}014=44493\,H$.

6. Additional research of the material

Linear density of the roving

A sample of roving with length of 1 m was weighed on Denver Instrument APX-60 (maximum permissible weight 60 g, measuring accuracy 0.1 mg.) Error in finding the length of specimen: ± 1 mm. Results are listed in table 9.

Table 9. Weight of 1 m roving.

№ weighing	1	2	3	4	5	6	7	8	9	**Average**
Weight, gr.	1.7214	1.7215	1.7212	1.7214	1.7215	1.7214	1.7214	1.7214	1.7215	**1.7214**

Figure 13. Specimen of the roving with length 1 m, subjected to weighting.

Linear density of roving, according to measurements - 1721.4 teks (according to the certificate (see table 1) - 1760 teks).

Measurement of the cross section of the roving in a free state

The measurement was carried out using a caliper. The following results were obtained: width ~ 5 mm, thickness ~ 0.3 mm. Diameter of twisted roving ~ 1 mm.

Photomicrography of the roving

The pictures were used to determine the diameter of the roving filaments. Based on the results of the measurements filament diameter is ~10 μm. It was noted the transparency of the filaments (Fig. 14).

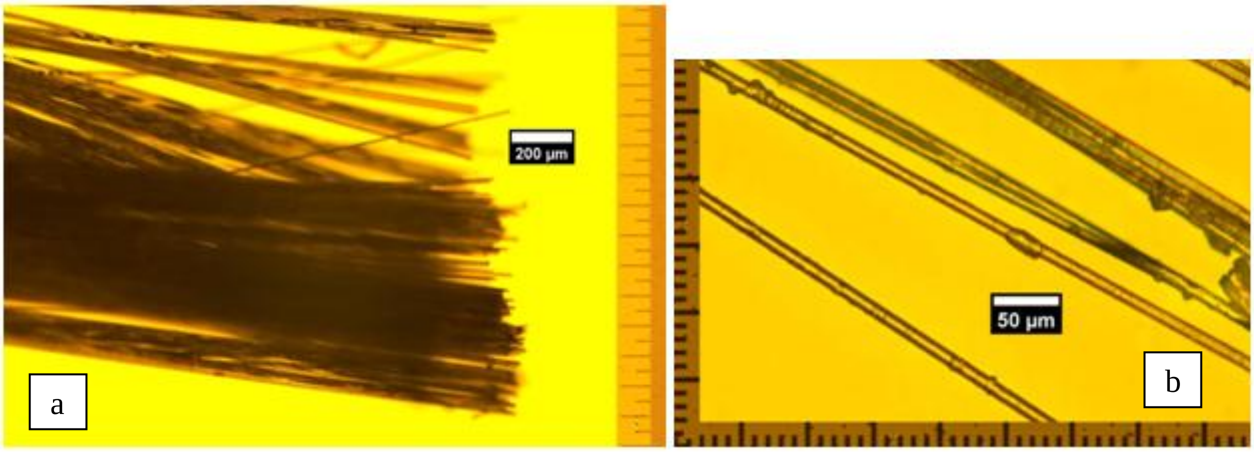

Figure. 14. Roving filaments (enlarged).

Thermal testing of the composite

After the destruction the elements of samples П1..П5 were subjected to half-hour exposure to different temperatures (Fig. 15). At temperature 250 ^{o}C composite slightly darkens, at 300 ^{o}C it darkens and begins to give off unpleasant smell, at 350 ^{o}C it emits strong smell and starts spilling out.

Figure. 15. Elements of the specimens after half an hour exposure excerpts to different temperatures.

Measurement of the cross-sectional area of roving

At dense packing of the roving with tension ~ 5 kg in the groove (cross section 19.2 × 3.1 mm) total number of strands of the roving amounted to 57 (fig. 16). Coming from here, the cross-sectional area of the roving in wrapped state - $\frac{19,2\times 3,1}{57}=1,044\ mm^2$.

Assuming that the cross section of the filament is 6-sided polygon of 10 microns diameter we would obtain cross-sectional area of the roving (1200 filaments) 0.416 mm^2 - most dense packing. In this case the groove could be filled up by 143 strands. The discrepancy between the practical and the theoretical density of packing is most likely be explained by real form of cross section of the filaments (fig. 14b), the presence of lubricant and insufficient stress tension during laying. For the complete straightening of the filaments of the roving applied tension should be 150...200 N (Fig. 11).

Note that average area per 1 roving of the specimens was at sections: (a), (б) - 1.36 mm^2; (c), (d) - 0.96 mm^2; (e), (f), (g), (h) - 0.92 mm^2 (Fig. 3).

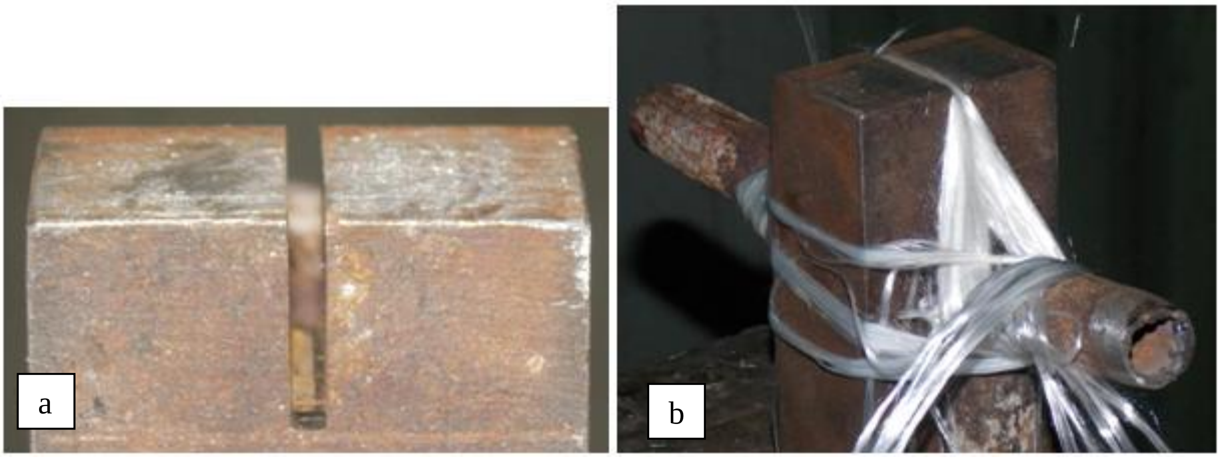

Figure. 16. Laying of roving in the groove: a - groove cross-section 19.2 × 3.1 mm; b - roving, laid into the groove.

7. Properties of the filler material

According to the data provided by MPRI NASB in the calculations, could be used the following (approximate) characteristics of filler:

-Young's modulus: 1.8-2.2 HPa;
-yield strength: 80 MPa;
-Poison’s ratio: 0.25
-impact strength 15 kJ/m^2.
-density: 1000-1100 kg/m^3.

8. List of references

1. TU UA 24.6-0030314547-002-2004- Epoxy resin КДА - ХИ. (ТУ У 24.6-0030314547-002-2004 - Связующее эпоксидное марки КДА - ХИ).
2. GOST 17139-2000-Fiberglass. Roving. Specification. Minsk. (ГОСТ 17139-2000 - Стекловолокно Ровинги Технические условия. Минск).
3. Gutnikov S. I., Lazorak B. I., Seleznyov A. N. Glass fibers: Handbook for students studying in composite nanomaterials. -M., 2010. -53 s. (Гутников С. И., Лазоряк Б. И., Селезнев А. Н. Стеклянные волокна : учеб. пособие для студентов по специальности «Композиционные наноматериалы». - М., 2010. - 53 с).
4. GOST 6943.10-79 - Textile glass. Method for determination of breaking load and elongation at break. (ГОСТ 6943.10-79 - Материалы текстильные стеклянные. Метод определения разрывной нагрузки и удлинения при разрыве.).