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MAIN STAGES FOR PRODUCTION OF CARBON FIBER TUBULAR RODS

Abstract: Carbon–fiber tubular rods (CFTR) are widely used in power structures of unmanned aerial vehicles and space vehicles. High-strength material allows to significantly facilitate the weight of structures. In this work, a study was made of the method for obtaining CFTR by the method of "wet" winding of a carbon roving impregnated with epoxy resin. The influence of the roving thickness on the tensile strength / contraction strength, the roving winding speed and effort, and the roving winding angle is studied. The maximum strength of CFTR was obtained at a roving thickness of 24K and the settings of a winding machine: roaming speed 18 mm /s, pulling forces 18.6N, cross-winding angle 55^0. The effect of processing "raw" windings in a vacuum bag at atmospheric pressure was obtained. Vacuum treatment reduces the porosity of the product and increases its strength. The tensile strength / compression strength of CFTR using epoxy resin with hardener hardening at room temperature was 346.5 MPa, at a temperature of 150 ^{0}C - 370 MPa, at a temperature of 180 ^{0}C - 516 MPa. It is assumed that the advantages of hot hardening of the epoxy matrix are due to its high fluidity, which allows penetration into all pores and to wet the carbon roving surface well.

Keywords: spacecraft, carbon-filled plastic, tubular rod, winding, winding machine, roving, epoxy resin, strength

Introduction. To date, for modern aerospace technology, there has been a steady trend of using composite materials, primarily carbon fibers. Carbon-filled plastic is a composite material consisting of a polymer matrix and a carbon fiber [1]. Due to unique properties, namely: high values of specific strength and rigidity, good resistance to alternating loads, low values of coefficient of linear thermal expansion, CFRP advantageously differs from traditional materials. Table 1 presents the comparative characteristics of structural materials [2].

Table 1 – Properties of structural materials

Material	Specific strength, м^2/с^2	Specific elasticity, 10^4 м^2/с^2	Relative elongation,%
Aluminum alloy AMg6	136	28	15-20
Aluminum alloy B 96	248	26	6
Titanium	355	24	8-20
Beryllium	432	162	4-12
Carbon fiber	up to 940	up to 193	0,5

As can be seen from Table 1, carbon fiber, in terms of strength characteristics and specific strength, surpasses other structural materials, which makes its use in space technology expedient.

In truss constructions of space design one of the main elements are tubular rods and rod systems, which are interconnected by couplings, hinges, fittings and various devices. Constructions of this type have high strength and stiffness characteristics, as well as a number of unique properties. This allows them to be used for structures of the supporting framework of space platforms, satellites, aircraft, telescopes, antennas and other objects operating under specific conditions [3]. Similar designs with a mass of up to 15 kg can withstand a destructive load of up to 900 kgf with a service life of at least 20 years. Using carbon -fiber elements allows you to save up to 50% of the total weight [4].

Figure 1 shows the carbon fiber truss structure of the space vehicle (hereinafter referred to as "SC") of the Yuzhnoye Design Bureau, [5]. This construction consists of rectangular carbon fiber pipes, which are connected together by titanium fittings.

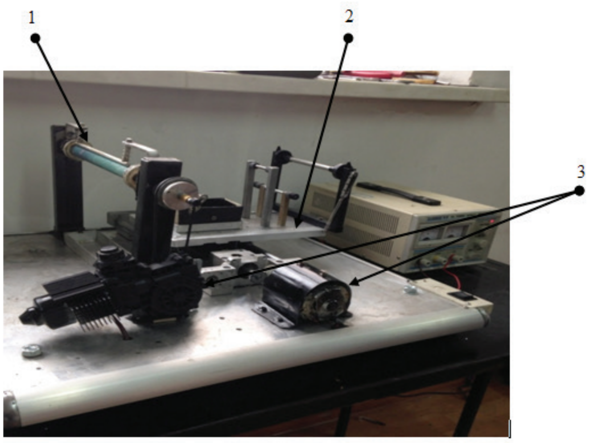
Figure 1 – Truss construction made of carbon fiber for space purposes

For the manufacture of carbon fiber tubular rods, there are many experimental methods. All these methods differ in winding technology. The paper [6] contains a description of the technology for spinning products from composite materials made of carbon fiber. Techniques of manufacturing differ conditionally on two types: “dry” and “wet” winding. In the “wet” winding process, the resin is applied to the carbon material during the winding itself. In the case of “dry” winding, prepregs are used. Prepreg is a composite material, a semi-finished product, a finished product pre-impregnated with binder and reinforcing materials [7]. It is believed that the method of “wet” winding gives the maximum strength characteristics of the sample, so this method is taken as the basis for research in the work.

This technology for the production of CFTR is intended for the production of experimental, later industrial, spacecrafts of JV “Galam” LLP, as well as university nanosatellites.

Aim of work is to develop a method for obtaining carbon fiber tubular rods by the method of “wet” winding of roving with competitive characteristics.

Experimental part and discussion of results. The most important technological parameters of the method of manufacturing carbon fiber tubular rods by the method of “wet” winding epoxy resin impregnated with carbon roving are: the thickness of roving, the speed of roving through the bath with epoxy resin (the time of roving in the resin), the tension of roving during winding, vacuuming, pressing and hardening shaped “raw” product. Due to these technological factors, little information has appeared in the literature. In view of this, experimental studies of these factors have become a necessity.



1 - winding core mandrel, 2 - movable carriage, 3 - electric motor

Figure 2 – Laboratory winding machine of design and construction of “National Center for Space Research and Technology” JSC

Manufacturing of CFTR samples. Manufacture of prototypes of carbon fiber tubular rods (CFTR) was carried out by “wet” winding of carbon roving on 2 laboratory winding machines: our design and manufacturing shown in Figure 2 and X-Winder (manufactured in USA, CNC), shown in Figure 3.

Both machines work constructively on the same principle. The winding mandrel 1, rotating from the electric motor 3a, stretches roving through a bath of epoxy resin. The movable carriage 2, moving from the drive of the electric motor 3b, along the winding mandrel sets the required winding angle. The braking system allows to adjust the force of the roving tension.

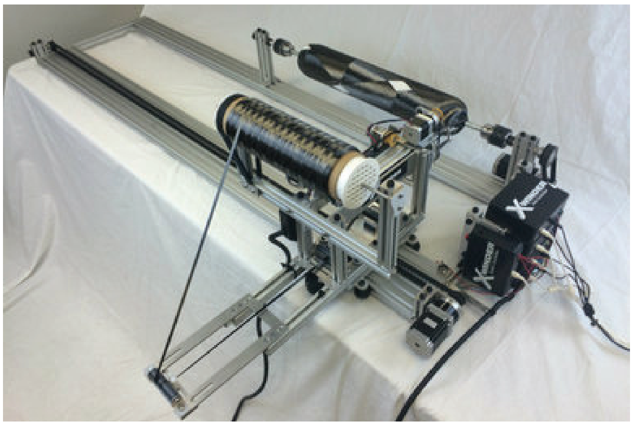


Figure 3 – Laboratory winding machine “X-Winder” (USA)

Roving (further-roving) and epoxy resin ED-20 with hardener PEPA, curing was carried out at room temperature. All components used for the preparation of carbon fiber are available on a free sale.

Strength tests of CFTR on tension-compression were carried out by preparing samples in accordance with State Standard 11262-80 (ISO R527). Figure 4 shows a standard for flat samples, by analogy with it, samples for testing CFTR were prepared. For the case of CFTR, the tube samples were calibrated from the ends of the winding of the fiberglass tape.

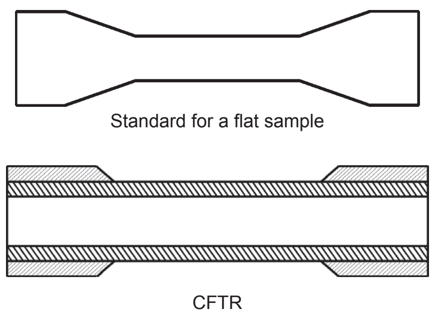


Figure 4 – Samples for testing tensile-compressive products

The effect of roving thickness on the strength of CFTR. The first step was to investigate the effect of the roving thickness on the quality of the produced CFTR. To this end, rovings with thicknesses of 16K and 12K were made from standard roving 24K thick (which we had in stock), and their general appearance is shown in Figure 5.

Samples of carbon tubular rods made of rovings with thicknesses of 24K; 16K, 12K with cross corners of winding 45^0. A sample of CFTR from roving 24K is shown in Figure 6

Figure 5 – General view of carbon rovings used in experiments

Figure 6 – CFTR from roving 24K with a cross-winding angle 45^0

The samples were tested for strength characteristics, density, mass ratio of roving and epoxy resin. The results of the tests are given in Table 2. The densest and most durable samples were obtained on roving 24K, for this case the proportion of reinforcement in the composite also turned out to be the largest. The reason for this should be found in the geometry of the weaving of the tube: a large roving leaves less free space filled with epoxy resin. However, the increase in the size of roving has limits, since too large roving will break the structure of the body of the tube and begin to give a loss of strength. A more detailed study of this question will allow ~~us~~ to find the optimum, this is the topic of a separate study. The received data allow choosing as the best variant for strength and geometry of a surface of tubes roving 24K. All further research was conducted with it.

Table 2 – Characteristics of CFTR from rovings of various thicknesses

№	Samples with different thicknesses	Density of samples, g / cm3	Ratio of roving and epoxy resin,%	Strength, MPa	
				compression	Stretching
1	12K	1,15	44:56	210	490
2	16K	1,21	42:58	234	500
3	24K	1,43	54:46	260	520

The nature of the destruction of CFTR was different from the nature of the destruction of epoxy resin. Epoxy resin is destroyed evenly in the center and at a time across the entire area (splits into pieces). Whereas CFTR is destroyed gradually (layerwise) along the direction of the carbon reinforcement. In the process of destruction, the epoxy resin is peeled off from roving, and microcracks are formed in the epoxy resin.

Influence of the speed of roving. The speed of roving in the bath with epoxy resin was regulated by changing the input voltage to the winding motor. The results of experiments with different roving pulling speeds are given in Table 3. Since the CFTR stretch-shrink experiments showed (Table 3) that the compressive strength is critical, then later the strength tests of the CFTR were limited to compression tests. The optimum speed of pulling the roving 24K for the bath of our winding machine (length 170 mm) was 18 mm / s or the time of roving 24K in epoxy resin was 9.5 seconds. The optimum is the balance of complex processes: rapid winding, which complicates the resin's impregnation of roving, and slow winding, leading to a thickening of the resin during the period of wet roving from the exit from the bath to the application to the winding rod. The result should be evaluated as the order of magnitude, since for each other winding machine, other brands of roving and epoxy will be other values of the optimum. However, the optimal amount of roving time in the resin bath will be about 9.5 seconds. It is interesting to note that at the work [8], a winding machine for fiberglass products with an optimal belt pulling speed of 23 mm / s was designed and used. This indicator was close to the indicator for our machine.

Table 3 – Characteristics of samples of CFTR with different threading speeds

№	Threading speed, mm/s	Roaming time in epoxy resin, s	Compressive strength, MPa
1	8	21,3	250
2	13	13,1	290
3	18	9,5	310
4	24	7,1	260

In subsequent experiments, the roving speed was 18 mm / s (roving time in epoxy resin 9.5 s).

Effect of roving pulling force. Further, the effect of roving tension on the strength of CFTR was investigated. The tension of the roving was regulated by belt braking of 2 rollers before the roving entered the bath and 4 rollers inside the bath with resin. The results of the experiments are given in Table 4. The best results were obtained for roving tension at 18.6 N winding (the maximum possible for the machine). For comparison, we give data for the winding machine [9], where the tension of the carbon roving was regulated within the limits of 17.8 - 26.7 N. The result obtained is close to the optimum, although it is advisable to increase the tension of the roving somewhat by performing a further modification of the machine. In subsequent experiments, the manufacture of CFTR was carried out with a roving tension of 18.6 MPa.

Table 4 – Characteristics of CFTR samples with different roving tension forces

№	Tension of roving, N	Compressive strength, MPa
1	3,5	190
2	7,8	230
3	11,4	310
4	18,6	330

Influence of the angle of winding roving. Unlike previous cases, there are a number of studies in the literature on the optimal angles of winding tubular rods from glass and carbon rovings [10-12]. These data give an optimal angle of cross-wound tubular rods for cases of tensile-compression axial load - 55^0. Due to the extremely important influence of this factor on the quality of CFTR, it was decided to manufacture and conduct tests of CFTR samples with cross-winding angles of 30^0, 45^0, 60^0, 80^0. The results are shown in Table 5

The data in Table 5 show that the sample at the winding angle 30^0 has a maximum tensile strength and a minimum compressive strength, for a sample with an angle of winding 80^0 all the way around. The greatest tensile-compression strength in our experiments was obtained on samples with a winding angle of 60^0 to 310 MPa (for comparison, the strength of market samples of CFTR is 120 MPa). The obtained data indicate that the optimal winding angle is in the vicinity of 600. This result is close to the above-mentioned literary data of other authors - 55^0. Perhaps the maximum in our case is also near 55^0, this will be checked during the overall optimization of the technology in 2017.

Table 5 – Dependence of the strength of CFTR on the angle of winding

Angle of rod winding, degrees	Compressive strength, MPa	Tensile strength, MPa	Compressive strength of market grades CFTR, MPa
30	230	520	120
45	270	450	
60	330	410	
80	380	280	

Influence of evacuation and pre-pressing of the "raw" molded product. An important factor affecting the strength of the material is its porosity. Figure 7 shows an optical image of the polished surface of epoxy resin ED-20, which was used in our experiments. Numerous gas bubbles are visible, which should give porosity, both epoxy and carbon fiber. In this connection, a common method of reducing the porosity of composites is to vacuum the "raw" preformed samples before the hardening operation. Vacuuming is necessary for sucking out of a "raw" product dissolved in epoxy resin gas, as well as air recruited during the operation of winding.

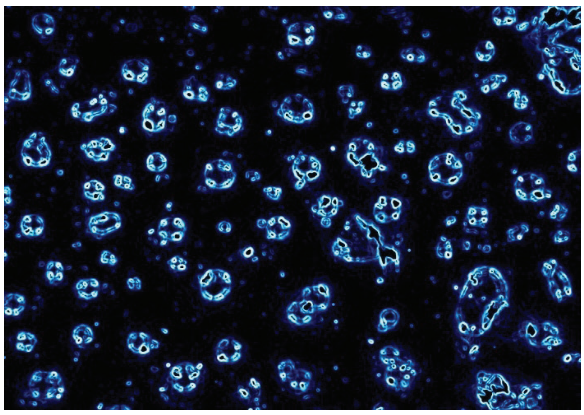


Figure 7 – Porous structure of cured epoxy resin ED-20 (200x)

Vacuuming is preferably carried out with the operation of pressing the product. These operations are well combined in the methods of processing a carbon fiber “raw” product by placing it in a vacuum bag, evacuation is accompanied by a sample under-pressing due to atmospheric pressure. The operation of vacuum processing is sometimes called “subforming”, when degassing is carried out under a vacuum of 0.04-0.01 MPa for at least 2 hours, at 15-30 ° C. In the process of aging, it is additionally suggested to periodically connect the cavity under a vacuum bag (bag) periodically for a few minutes with the atmosphere and re-create a vacuum. Further, the curing process is carried out [13]. Figure 8 shows the process of “sub-molding” of CFTR.

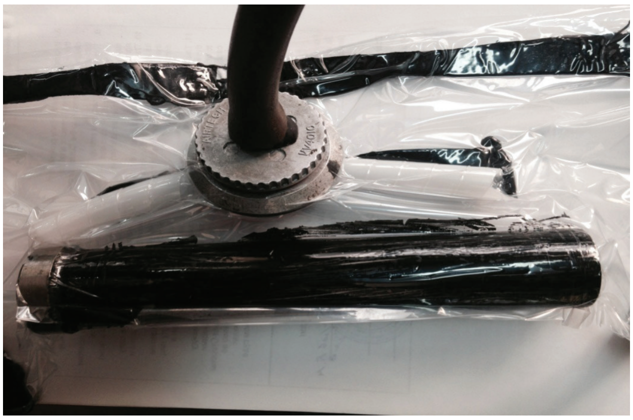


Figure 8 – Vacuuming process of the formed «raw» CFTR in a vacuum bag

The sample was hardened at room temperature. Samples that passed “sub-molding” gave a compression strength of 346.5 MPa, i.e. 5 % higher than samples without vacuum degassing and pre-pressing. The necessity of this operation becomes obvious. In all subsequent experiments, the “sub-molding” operation was included in the list of mandatory technological operations for the manufacture of CFTR.

Effect of a matrix of hot hardening. In the experiments, two matrices with different hot hardening temperatures:

a) ED-20 resin with iso-MTPA (methyltetrahydrophthalic anhydride) hardener in a weight ratio of 100 g of resin per 85 g, hardening according to the program 100 ^{0}C/1 h + 120 ^{0}C/3 h + 150 ^{0}C/7 h [14]

b) Ethanol-Inject-T resin (component A) with a hardener (component B) at a mass ratio of 100 : 49.9 %, hardening according to the program 150 ^{0}C/4 h + 180 ^{0}C/1h [15]

Samples of CFTR were prepared according to the above full scheme. CFTR, manufactured with the matrix of option a), had the following strength characteristics: a tensile strength of 700 MPa; At a compression of 370 MPa. CFTR with a matrix in variant b) showed a tensile strength of 730 MPa, a compression of 516 MPa. Thus, the resin with the highest temperature of hardening, Etal-Inject-T (180 °C), gave the highest strength of CFTR. The increase in the strength of CFTR for compression compared with the pair ED-20 + PEPA (25 °C) was 49 %, and for the ED-20 + iso-MTPA (150 °C) - 6.7 %. This very important result is explained by the fact that the higher the epoxy hardening temperature, the higher its fluidity, its ability to penetrate all micropores of carbon filaments and roving, which increases the adhesion of the matrix to the carbon reinforcement.

Conclusions. The best results on the strength of CFTRs on tension-compression are obtained using roving with a thickness of 24K and the following modes of the winding machine: the speed of roving in a bath with epoxy resin of 18 mm/s (roving time in epoxy resin 9.5 s), the tension of roving 18 , 6 N, angle of winding roving 55^0.

Vacuuming the “raw” molded CFTR in a vacuum bag at atmospheric pressure gives an increase in strength of about 5 %.

Epoxy resins of hot hardening give hardening to CFTR. Thus, the maximum value of the compressive strength of CFTR for the solidification of the matrix at room temperature (ED-20 + PEPA-polyethylenepolyamine) was 346.5 MPa, for the solidifying matrix at 150 ^{0}C (ED-20 + iso-MTPA) - 370 MPa, for the matrix of hardening at 180 °C (iso-MTPA with its hardener) - 516 MPa. Obtained experimental samples of CFTR significantly exceed the strength of market samples of CFTR, which are of the order of 120 MPa.

It is assumed that the advantages of hot hardening of epoxy are due to its high fluidity, which allows penetration into all pores and to wet the surface of carbon roving.

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ТҮЙІНДЕМЕ

Ғарыш және басқарусыз ұшатын аппараттардың ауыр конструкцияларында көміртекті түтікшелі білекшелер (КТБ) кеңінен қолданылады. Жоғары берікті материалдар конструкция массасын біршара төмендетеді. Жұмыста КТБ көміртекті ровингтің эпоксид шайырымен «ылғалды» орау әдісі арқылы алудың жолдары зерттелген. КТБ созу/қысу беріктігіне қатысты ровингтің орау бұрышының, кернеу және жылдамдығының, ровингтің қалыңдығың әсерлері зерттелді. КТБ максимальды беріктігі ровингтің 24К қалыңдығы кезінде және орау қондырғысының мынадай сипаттамалар: 18 мм/с ровингтің жылдамдығы, 18,6 Н тежеу кернеуі, 55^0 орау бұрышы негізінде алынды. Вакуумдық қап арқылы атмосфералық қысымда өңдеу әсері зерттелді. Вакуумдық өңдеу бұйымдағы пор санын азайтуға және беріктігін арттыруға әсерін тигізеді. КТБ созу/қысу бойынша беріктігі бөлмелік температурада

қатаю бойынша 346,5 МПа, 150^{0}С температурада – 370 МПа, ал 180^{0}С температурада 516 МПа көрсетті. Эпоксид шайырының жоғары температуралы қатаю, оның жоғары сұйықтығында порлардың ішіне кіруімен және көміртекті ровинг бетінің жақсы адгезиялауымен түсіндіруге болады деп түсіндіріге болады

Түйінді сөздер: ғарыш аппараттары, көмірпластик, түтікшелі білекше, орау, орау қондырғысы, ровинг, эпоксид шайыры, беріктік

РЕЗЮМЕ

Углепластиковые трубчатые стержни (УТС) широко используются в силовых конструкциях беспилотных летательных и космических аппаратов. Высокопрочный материал позволяет существенно облегчать массу конструкций. В работе проведено исследование метода получения УТС методом «мокрой» намотки углеродного ровинга, пропитанного эпоксидной смолой. Исследовано влияние на прочность на растяжение/сжатие УТС толщины ровинга, скорости и усилия намотки ровинга, угла намотки ровинга. Максимальная прочность УТС получена при толщине ровинга 24К и параметрах настройки намоточного станка: скорости протяжки ровинга 18 мм/с, усилия протяжки 18,6Н, угла перекрестной намотки 55^0. Получено влияние обработки «сырых» намоток в вакуумном мешке при атмосферном давлении. Вакуумная обработка позволяет снизить пористость изделия и повысить его прочность. Прочность УТС на растяжение/сжатие с использованием эпоксидной смолы с отвердителем, твердеющих при комнатной температуре, составила 346,5 МПа, при температуре 150 ^{0}С – 370 МПа, при температуре 180 ^{0}С – 516 МПа. Сделано предположение, что преимущества горячего твердения эпоксидной матрицы, обусловлены ее высокой текучестью, позволяющей проникать во все поры и хорошо смачивать поверхность углеродного ровинга.

Ключевые слова: космические аппараты, углепластик, трубчатый стержень, намотка, намоточный станок, ровинг, эпоксидная смола, прочность

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OBTAINING CALCIUM-PHOSPHATE COATINGS ON TITANIUM SUBSTRATE UNDER CONDITIONS OF MICRO-ARC OXIDATION

Abstract: The results of experiments on microarc oxidation of a substrate of titanium grade VT 1-0 under conditions of anodic treatment in phosphoric acid electrolytes containing calcium ions at a pH of 1 to 7 and a current voltage of 150 to 250 V are presented. The coatings were investigated by scanning electron microscopy, X-ray phase analysis and optical microscopy. The structure, phase and chemical composition formed as a result of micro-arc treatment of coatings is described. As a result of the studies, optimal regimes and parameters for obtaining calcium-phosphate coatings were established and determined. Processing with the modes found allows one to obtain coatings consisting of a mixture of phases Ca0.5(Ti2P3O12), CaTi4(PO4)6, Ca(PO3)2 and Ca2P2O7, which, according to the literature, are biocompatible compounds. The results of the SEM surface of the obtained coatings showed the presence of three structural components: sponge aggregates in the form of honeycombs, large bubbles having one or more shells, dense lenticular plates. The atomic ratio in the calcium-phosphate coatings varied in the range 0.30-0.62. It is shown that by varying the pH solutions and the magnitude of the stress of the microarc machining process, it is possible to significantly affect the structure, phase composition and thickness of the coatings produced. Promising from the point of view of obtaining biocompatible coatings is microplasma anodic treatment of titanium in phosphate acid electrolytes at pH ~ 3 – 1. A conclusion was made about the prospects of processing endoprostheses from titanium alloys by this method, to improve their coalescence with bone tissue.

Keywords: biocompatible materials, implant, crystallization, microarc oxidation, bioresorption, calcium-phosphate coatings

Introduction. Titanium (Ti) and its alloys are widely used as materials for the manufacture of surgical implants because of their excellent mechanical properties, high resistance to corrosion, low specific gravity and good biocompatibility [1, 2]. However, since the surface of titanium does not contribute to ostiointegration, special biocompatible coatings are used to ensure adhesion to bone tissue [3]. Calcium-phosphate coatings (CF) provide both biological activity and osseointegration. Among them, hydroxyapatite of calcium (GA) ($Ca_{10}(PO_4)_6(OH)_2$) attracts the greatest attention for clinical use due to its close