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THE EFFECT OF CARBON FABRICS MODIFICATION ON THE STRENGTH OF CARBON FIBER REINFORCED PLASTIC

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Abstract. This article reviews the study of influence of carbon fabrics modification on the strength of carbon fiber reinforced plastic. Two types of epoxy compound Etal Inject SLM of “cold curing” and Etal Inject-T of “heat curing”, nitric acid HNO₃ of concentration 60 %, carbon fabric of Toho Tenax/Aksa 3k-1200-200 were used in the study. Comparisons of the strength properties of carbon plastic on these compounds were given. The best result was obtained on Etal Inject-T with tensile strength – 1000 MPa and in compression – 425 MPa. Carbon fabric modification was carried out by grafting carboxylated groups treated with HNO₃ to the carbon fiber surface. The treatment time in acid varied from 0.5 to 6 minutes. The compressive strength by 17 % from 425 MPa to 497 MPa has been established to increase when carbon fiber by HNO₃ is modified for from 0.5 min to 2 min, then the strength decreases enlarging the treatment time. The decrease in strength is associated with a supersaturation of the surface of the fibers with carboxyl groups, which were destroyed during heat treatment. Thus, the surface oxidating of carbon fabric is the most effective method of increasing its adhesion to epoxy resin and the strength of carbon fiber. The functional groups formed during the oxidation process allow for a tight cross-linking of the epoxy matrix with carbon fiber. It was previously found that the strength of carbon fiber increases with the modification of epoxy resin with oxidized carbon nanotubes. An assumption is made about a uniform mechanism for strengthening carbon-fiber reinforced plastic due to chemically active functional groups, both on the surface of carbon fiber and on the surface of carbon nanotubes.

Keywords: carbon fiber reinforced plastic, carbon fabrics, modification, strength.

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КОМІРҰЛПАНЫ ЖЕТІЛДІРУДІҢ КОМІРПЛАСТИК БЕРІКТІЛІГІНЕ ӘСЕРІ ТУРАЛЫ

Түйіндеме. Мақалада, көміртеқпластик беріктік қасиетіне көміртек матасының модификациясының әсерінің зерттеулері келтірілген. Екі түрлі эпоксидті компаунд – «суық қатайтылған» Этал Инжект SLM және «ыстық қатайтылған» Этал Инжект-Т, 60 % қоюланған азот HNO₃ қышқылы, Toho Tenax/Aksa 3k-1200-200 маркалы көміртегі кенеп матасы пайдаланылды. Аталған компаундтардағы көмірпластиктің беріктік қасиеттеріне салыстыру жүргізілді. Ең жоғары нәтиже 1000 МПа созу кезінде және 425 МПа қысу беріктік шектеріндегі Этал Инжект-Т алынды. Көмірұлпаны жетілдіру көміртегі талшықтарының бетіне HNO₃ өңделген карбоксилденген топты егу жолымен іске асырылды. Қышқылда өңдеу уақыты 0,5 минуттан 6 минутқа дейінгі аралықта жалғасты. HNO₃ көміртегі талшығын 0,5 минуттар 2 минутқа дейін жетілдіру кезінде қысуға беріктілік шегі 17 %-ға, 425 МПа-дан 497 МПа-ға дейін артатыны анықталды, әрмен қарай уақытты арттырған сайын беріктілік төмендей бастайды. Беріктік қасиетінің төмендеуі талшықтардың карбоксилді топтармен қанығуымен байланысты яғни термиялық өңдеу кезінде бұзылғандықтан. Осылайша көміртеқматаны тотықпен өңдеу үрдісі, эпоксидті шайырға адгезиясын жақсартып көмірпластиктің беріктігін арта түсіреді. Эпоксидті матрицасымен көміртек талшықтарды тығыз байланысқа осы белсенді топтар мүмкіндік береді. Эпоксидті шайырдың модификациялау кезінде көмірпластиктің беріктігі ұлғаятыны расталды, яғни көміртек талшықтар беттері мен нанотүтікше беттеріндегі химиялық белсенді топтар арқасында.

Түйін сөздер: көмірпластик, көміртегі талшығы, жетілдіру, беріктілік.

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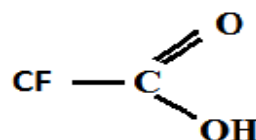
ВЛИЯНИЕ МОДИФИКАЦИИ УГЛЕТКАНИ НА ПРОЧНОСТЬ УГЛЕПЛАСТИКА

Резюме. В статье представлены исследования влияния модификации углеткани на прочность углепластика. Использовался эпоксидный компаунд двух видов – Этал Инжект SLM «холодного отверждения» и Этал Инжект-Т «горячего отверждения», азотная кислота HNO_3 концентрации 60 %, углеродное полотно марки Toho Tenax/Акса 3к-1200-200. Приведены сравнения прочностных свойств углепластика на этих компаундах. Лучший результат получен на Этал Инжект-Т с пределами прочности на растяжение – 1000 МПа и на сжатие – 425 МПа. Модификацию углеткани осуществляли путем привития к поверхности углеродного волокна карбоксильных групп, обработанных в HNO_3 . Продолжительность обработки в кислоте варьировали от 0,5 до 6 мин. Установлено, что при модификации углеродного волокна HNO_3 в течение от 0,5 мин до 2 мин предел прочности на сжатие возрастает на 17 % с 425 МПа до 497 МПа, далее с увеличением времени обработки прочность снижается. Снижение прочности связано с перенасыщением поверхности волокон карбоксильными группами, которые при термической обработке разрушаются. Таким образом, показано, что окислительная обработка поверхности углеродной ткани является наиболее эффективным методом повышения ее адгезии к эпоксидной смоле и прочности углепластика. Функциональные группы, образованные в процесс окисления позволяют обеспечить плотную сшивку эпоксидной матрицы с углеродным волокном. Подтверждено, что прочность углепластика увеличивается при модификации эпоксидной смолы за счет химически активных функциональных групп, как на поверхности углеродного волокна, так и на поверхности углеродных нанотрубок.

Ключевые слова: углепластик, углеродное волокно, модификация, прочность.

Introduction. CFRP is widely used as engineering materials (EM). A combination of properties such as high specific strength and rigidity, high wear resistance and resistance to aggressive media differ them from standard EM. Hardening of CFRP is known to be carried out by modifying epoxy resin, carbon fiber (CF) or their interphasal activity [1, 2]. We investigated the effect of surface modification of carbon fiber by oxidation on the strength characteristics of CFRP of the three methods of hardening. Assuming the fact that the surface of carbon fiber is chemically inert and has insufficient adhesion, therefore, adversely affect the strength of CFRP. To ensure a strong adhesive bond between carbon fiber and polymer, it is necessary to oxidize them in order to bring to the surface, for example, oxygen-containing surface complexes, which are formed as a result of oxidative treatment, either in the gas phase or in the solutions. The treatment gives three types of superficial oxides: acidic, basic and neutral. Treating with different oxidating solutions is one of the methods of distribution of acidic superficial oxides (carboxyl and carboxyl-carbonate, phenolic and lactone groups) to the surface [3-5].

The method of functionalization of carbon nanotubes is equivalent to the oxidation of carbon fibers [6]. As a rule, oxygen-containing acids and based on them mixtures are most commonly used as oxidizing agents: HNO_3 , $\text{HNO}_3 + \text{H}_2\text{SO}_4$ etc. As a result of this treatment, depending on the type of an oxidant, carboxylic acids — COOH , carboxylic hydroxy acids — COOH and OH groups are activated on the surface of the carbon fiber [7]. Carboxyl and hydroxyl groups are formed on the surface in the process of treatment with a mixture of acids $\text{HNO}_3 + \text{H}_2\text{SO}_4$. Carbon fiber modification with nitric acid is written as:



Adhesion at the interface of the carbon fiber-polymer matrix is determined by the following factors: 1) chemical bonds between the surface of the carbon fibers and the polymer matrix; 2) physical bonds due to van der Waals forces, which are less durable than chemical bonds. The chemical interaction of the modified CF with epoxy resin without a hardener can be written in the following form [8-10].

As provided by the above chemical reaction, the CFRP production treated in HNO_3 consists of three stages:

- 1) carbon fiber activation by COOH group cultivation on the surface (when treating with nitric acid, carboxyl groups are formed)
- 2) carbon fiber secondary activation (new bonds formation) with epoxy when placed modified carbon fiber in a liquid epoxy resin without a hardener;
- 3) interaction with the epoxy group and the formation of a cross-linked polymer by adding a hardener to the epoxy resin.

Search by references revealed only a few papers to the subject under discussion, since there were few studies regarding the modification of the carbon fiber surface treatment with nitric acid.

The authors [11] carried out the carbon fiber oxidation by HNO_3 when heated (90°C) for 1.5 h, after processing the surface of the fiber became more rough and the oxygen concentration increased significantly after surface treatment, which improved the adhesion between the fiber and the matrix. In [12], the surface of carbon fibers was

changed using the method of chemical modification with nitric and hydrochloric acids. Analysis of the average tensile strength of carbon fibers of the original and treated with hydrochloric and nitric acids, shows that the strength values decreased with an increase in the oxidation time from 5 to 20 minutes (from 2143 MPa to 1531 MPa). However, this property was found to be significantly reduced in samples treated with nitric acid than in treated with hydrochloric acid, which is explained by the oxidizing properties of the nitric acid.

In [13], the effect of modified carbon fiber on the strength of CFRP was studied, the data are provided in Table 1. Reviewing Table 1, the acidic treatment of carbon fiber in a solution of nitric acid at its boiling point makes it possible to increase the shear strength of CFRP in 1.5-2 times. Enlarging the time of carbon fiber processing diluted with HNO₃ leads to the strength of CFRP increases continuously; in case of treating with concentrated

HNO₃, the strength passes a maximum level. However, these data do not determine the appropriate mode of carbon fiber treatment in the nitric acid, which gives carbon fiber maximum strength.

References of [11–13], provide the modification significantly affects the mechanical properties of carbon fiber and CFRP. However, these works do not provide a more detailed description with an indication of the comparative data of the hardening efficiency of various types of carbon modification on the strength of carbon fiber, there are no specific data about the compositions of modifying additives, the conditions of their application.

Considering the fact that the production of carbon fiber has no unique technological standard, due to the raw material and technological differences, the data from different authors is obtained with differing strength characteristics.

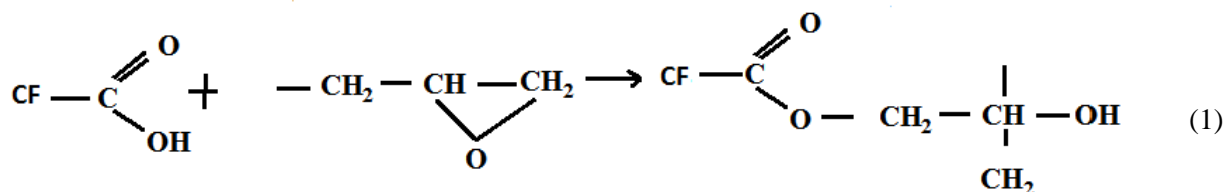


Table 1 - The effect of carbon fiber treating by nitric acid on the fiber and CFRP properties [13]

Acid	Treatment time	Fiber properties			The CFRP strength in shear, MPa
		Specific surface, m ² /g	Roughness factor	The content of COOH-groups x10 ² , wt.%	
Not treated		0,40	1,21	0,64	38
Diluted	6	0,62	1,88	-	54
	24	0,71	2,16	-	78
High concentration	0,5	1,21	3,67	-	77
	1,0	1,20	3,64	1,0	68
	6	1,03	3,27	1,7	50
	15	1,20	3,64	1,7	55
	24	1,03	3,12	1,85	54

The process optimization and the study of oxidative treatment of carbon fiber are obviously of interest in order to control the surface, increase the adhesive interaction of the carbon fiber with the epoxy resin, and the strength of CFRP. However, these issues are actually not investigated. *This study is aimed* to a testing study of the oxidizing process

of surface treatment of carbon fiber by the nitric acid and the determination of the appropriate composition of the epoxy resin to increase the strength of CFRP.

Testing and the methods of research. To find the solution to the task, the CFRP patterns were shaped as plates without modification and with modification of CF. The main components of CFRP

are carbon fabric, epoxy resin and its hardener. These elements form a set of basic CFRP characteristics. To obtain CFRP plates, the following components were used: 1) matrix of plates: epoxy compounds Etal Inject SLM of "cold" curing and Etal Inject-T of "hot" curing [14, 15]; 2) reinforcing components of the plates: carbon fabric - Twill 2/2 3K-1250-200 of twill weave.

In [6], a description of the CFRP production, modified CNTs of the Taunit-M brand (TS 2166-001-02069289-2006, LLC NanoTehCenter) is provided. Modified CNTs were introduced into the liquid ER and mechanically stirred was dispersed by ultrasonic treatment for 1 hour.

A solution of HNO_3 was used to modify the CF surface. The percentage and density of HNO_3 was carried out using a hydrometer.

When measured by hydrometer, the density of nitric acid was 1369 kg/m^3 , which corresponds to 60% HNO_3 .

The process of modifying carbon fabric patterns was carried out with a slow shutter speed in 60% HNO_3 solution for 0.5 to 6 minutes. After which the fabric was repeatedly washed with distilled water and dried at 110°C for 1.5 hours.

Under experiments to produce carbon fiber plates the method of manual molding was applied with mechanical pressing. The process of manual molding consists of several stages:

1 A laying form is prepared where the glass plate was used. A separating layer was applied to the laying form, which allows, later, to separate the blank. Separating alcohol of Loctite 330 NS brand was used as a separating layer.

2 Carbon fabric of the required size is laid out in layers into the form.

3 A binder component is prepared (when preparing the Etal Inject SLM compound, the components of the compound are mixed at room temperature, and the Etal-Inject-T compound is heated at 45°C for 60 minutes before mixing).

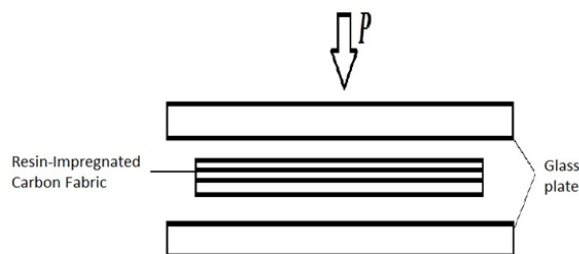
4 Each layer is impregnated with epoxy resin using a brush or soft roller (Figure 2 a).

5 At the final stage, the CFRP is rolled using a stiff roller: rolling eliminates air bubbles in the material.

6 CFRP was cured under pressure of $P = 0.02 \text{ MPa}$. A plate with a load was used to apply pressure (figure 2b).



a



b

a - fabric impregnation with the epoxy resin; b - CFRP curing under pressure

Figure 2 - Manual molding followed by CFRP pressing

Removing the product from the matrix was carried out only after complete curing, the curing time of the plates depends on the type of epoxy resin used. When using Etal Inject SLM epoxy compound, the CFRP cures at room temperature. The plate curing with epoxy resin of Etal Inject-T brand was carried out using the following temperature regime: 4 hours at 150°C and 1 hour at 180°C . To cure CFRP at high temperatures, a SNOL brand heat chamber with a heating temperature of up to 220°C was used.

To identify the strength characteristics of CFRP made of two types of resins, tensile and compression tests were carried out. Tensile testing

patterns were manufactured according to the international standard ISO R527 [16, 18, 19, 20]. Tests of CFRP tensile and compression patterns were carried out using a universal Shimadzu testing machine with the Trapezium X embedded software.

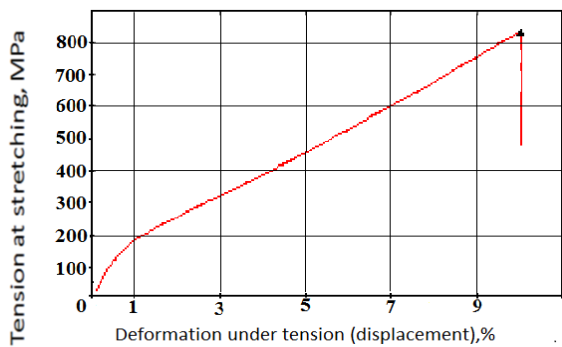
The tensile patterns made of carbon fabric and epoxy resin of two brands (Etal Inject SLM and Etal Inject-T) have a width of $15 \text{ mm} \pm 0.5 \text{ mm}$, a total length of 250 mm, and a thickness of 2 mm. The adhesion area for gripping on a bursting installation is 50 mm, the surface of this zone, for better grip, is treated with emery paper to a lustreless condition. CFRP patterns for compression tests are made with dimensions: width $20 \text{ mm} \pm 0.5 \text{ mm}$, total length 60 mm and thickness 3.2 mm.

The results and discussion. As a result of tensile and compression tests, the strength of CFRP patterns data were obtained with two types of matrix. To identify the average strength of CFRP, serial (repeated) tests (from three to five) were carried out.

Figure 3 shows the results with the average tensile test for CFRP patterns with Etal Inject SLM matrix (Figure 3a) and Etal Inject-T (Figure 3). The tensile strength properties of carbon fiber made of epoxy compound Etal Inject T are 20% are seen to be higher than when using Etal Inject SLM resin.

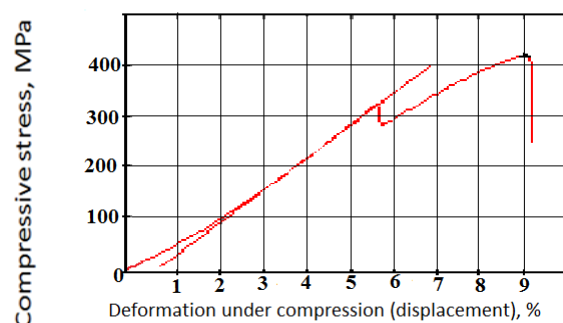
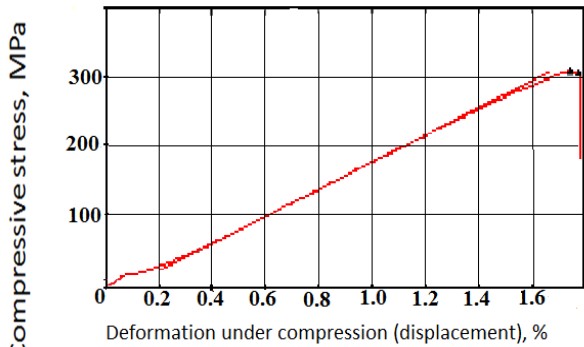
The ultimate strength at failure of a pattern with Etal Inject-T is 1000 MPa with a relative deformation of 5.5%, and the complete destruction of a CFRP patterns with epoxy matrix Etal Inject SLM occurs at a relative elongation of 10%, the maximum tensile stress is 833 MPa.

When testing the plates for compression, the strength characteristics of the patterns with the etal epoxy matrix Etal Inject-T were also higher than those of the patterns made of epoxy resin Etal Inject SLM. Figure 4 shows the results of the compression tests.



a - Etal Inject SLM patterns; b - Etal Inject-T patterns

Figure 3 - Tensile strength of CFRP patterns using two types of epoxy compound, untreated carbon fabric



a - Etal Inject SLM patterns; b - Etal Inject-T patterns

Figure 3 - Compression strength of CFRP patterns using two types of epoxy compound, untreated carbon fabric

The strength properties of compressed CFRP made of epoxy compound Etal Inject-T are found to be 30% higher than when using Etal Inject SLM resin. The tensile strength at failure of a pattern with Etal Inject-T is 425 MPa with a relative deformation of 8%, and the complete destruction of CFRP pattern with epoxy matrix Etal Inject SLM occurs at a relative deformation of 10%, the maximum stress is 300 MPa. The test results show the effect of the matrices used on the strength of the CFRP composite. High strength properties of the

composite were obtained using etal epoxy compound Etal Inject-T. Therefore, it is preferable to use the compound of “hot curing” Etal Inject-T, than when using epoxy resin Etal Inject SLM. When laying and high temperatures of curing up to 150 °C, the resin retains its viability, and the viscosity of the resin decreases (becomes more liquid), thereby impregnating of carbon fabric with the resin improves, these factors positively affect the uniformity and quality of the composite. Since, when conducting tensile and compression tests, Etal

Inject SLM matrix patterns were inferior in strength indicators to the patterns made from Etal Inject-T epoxy binder, Etal Inject-T patterns were used for further serial tests. Table 2 provides the results of the testing studies of the strength characteristics of the CFRP as a function of the exposure time of the carbon fabric in the HNO₃ solution.

As the data provided in Table 2, when the appropriate processing time of CF by HNO₃ is seen to be exceeded, the strengthening effect on the CFRP drops sharply. This effect arises an explanation that at a pausing 4 and 6 minutes there is a supersaturation of the surface of the fibers with carboxyl groups, which are destroyed during the heat treatment (drying). As a result, impregnability of carbon fabric with epoxy resin deteriorates and the material exploitation decreases sharply. In our opinion, this effect requires further study.

Table 2 - Effect of modification time on the strength of CFRP

Curing time, min	Compression strength, MPa
No modification	425
0,5	443
1	462
1,5	480
2	497
2,5	475
3	450
3,5	434
4	395
4,5	390
5	387
5,5	382
6	380

The effect of the CF modification efficiency is achieved at a pausing for 2 min in HNO₃, the strength of CFRP increases by 17% from the initial pattern, from 425 to 497 MPa.

Consider this pattern as an example, to follow the effect of the CF modification on the stress-strain state of the CFRP.

Figure 5 provides the curve of the stress-strain state of the CFRP have a linear temper up to destruction. Throughout the curve, the slope of the dependence $\sigma(\epsilon)$ increases so far as compression, i.e., the elastic modulus E increases. The characteristic rate of deformation degree was in the range of 10-11% in all cases. A similar effect of

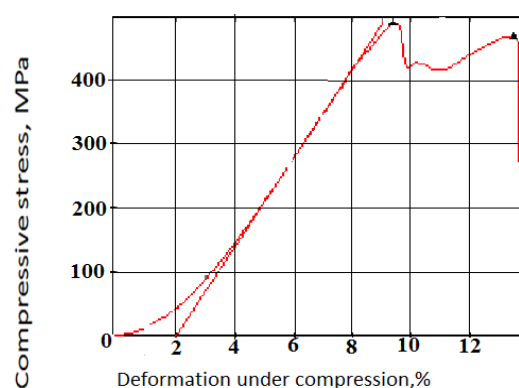


Figure 5 - Dependence of the CFRP compression strength with the modified CF in HNO₃ for 2 min

CFRP reinforcement was found during the modification of CNT, as provided in Table 3 [17].

As provided in Table 3 the original CNTs are seen to have no affect on the compression strength of CFRP, the hardening is obtained by functionalized CNTs. The introduction of a modified CNT gives hardening from 410 MPa to 426 MPa, the modulus of elasticity E from 14.1 GPa to 16.3 GPa. Consequently, the oxidation of the surface of CF or CNT is the most effective method of increasing their adhesion to epoxy resin and strength. Functional groups allow for a tight cross-linking of the epoxy matrix with the CF.

Table 3 Strength properties of CFRP, modified CNT [17]

Pattern	Content, %	$\epsilon_B, \%$	σ_B, MPa	E_B, GPa
CFRP	-	2,9	410	14,1
Initial CNT	0,05	-	410	-
	0,1	-	409	-
	0,15	-	409	-
Carboxylated CNT	0,05	2,6	417	16,0
	0,1	2,6	421	16,1
	0,15	2,6	426	16,3

Findings. The effect of two types of Etal Inject SLM and Etal Inject-T matrices on the strength properties of CFRP has been studied. The best result was obtained on Etal Inject-T. The increase in the tensile strength of CFRP was: 1) in tensile is 20% from 833 MPa to 1000 MPa, 2) in compression is 30% from 300 MPa to 400 MPa.

The modes of carbon fabric modification in the time range from 0.5 to 6 min with a HNO₃ solution for the strength of CFRP are investigated.

The highest rate of hardening was obtained at a two minutes processing time. Compression strength of CFRP is increased by 17% from 425 MPa to 497 MPa. An assumption is made about a single mechanism for strengthening CFRP due to chemically active functional groups both on the surface of carbon fiber and on the surface of CNTs.

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