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The features of chemo-mechanical activation technology of polymer composite materials production

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The paper deals with the study of the processes of obtaining polymer composites by chemo-mechanical activation technology (CMA-technology). The methods of research and testing of composite materials based on polytetrafluoroethylene (PTFE) and carbon fibers are analyzed and generalized. The influence of CMA technology on the structure and properties of PTFE composite was determined.

Keywords: polymer composite, polytetrafluoroethylene, carbon fibers, chemo-mechanical activation technology.

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Introduction

Nowadays, the production of new polymer composite materials with improved performance properties is crusial [1]. Fluorine-containing materials are among the most popular in various industries [2].

The object of study of this work is the technology of obtaining composites based on polytetrafluoroethylene and carbon fibers. The use of PTFE as a matrix affects the antifriction properties of the resulting composite materials.

PTFE composites are widely used due to the unique characteristics of polytetrafluoroethylene. Among its advantages are high heat resistance, high friction coefficient, the highest chemical resistance among thermoplastics, the ability to be used at low temperatures, and durability [3].

Among all the fillers, carbon fibers are the most effective because of their mechanical properties and light weight [4]. The use of carbon fibers significantly improves the wear resistance and increases the heat resistance of the composite material.

Generally, according to already known technologies, to obtain composites polytetrafluoroethylene powder is mechanically mixed with fillers, followed by pressing and sintering. However, this method has a number of disadvantages due to the high melt viscosity of PTFE. Therefore, it is reasonable to develop the technology of consecutive chemo-mechanical activation of PTFE and carbon fiber samples before their mixing [5].

Technological progress shows that the use of chemo-mechanical activation technology is promising for improving the antifriction properties [6, 7] of polymer composite materials, as it increases their toughness, stiffness, thermal conductivity, and heat resistance, which increases the reliability and durability of products made from such materials.

According to the CMA technology, carbon fiber was pretreated with a 20 % aqueous solution of flame retardants, annealed at high temperatures (723-2673 K), surface intermediate compounds were removed, pressed, kept at a special temperature, conditioned, crushed, and mixed with polytetrafluoroethylene at ultra-high speeds (5000-25000 rpm) in crushers, mills, dismembrators, or disintegrators [8]. At the last stages, the PTFE composite blanks were pressed and sintered.

For the mechanical activation of carbon materials, we used a crusher KDU-2.0 "Ukrainka" (with a knife speed of n = 1440 rpm), a crusher MRP-1 (with a knife speed of n = 7000 rpm), and MRP-1M (with a knife speed of n = 7350 rpm). The MPP-1 and MPP-1M were used for ultrafine grinding of the components and mixing the composition [9].

I. Technological parameters for matrix preparation and modification

Polytetrafluoroethylene (PTFE-4) with fibrous, spherical, and scaly particle shapes was used for the study.

First, PTFE blanks obtained by pressing are molded in the cold and then sintered in an electric furnace at 365-385 ^{0}C. This helps to harden the polymer and increases its strength under friction.

PTFE samples obtained by pressing and injection molding have the following physicomechanical properties at room temperatures [10]: density 2150-2220 kg/m^3, tensile strength 14-35 MPa, bending strength 14-18 MPa, compressive strength 10-12 MPa, elongation at break 250-500 %, compressive elastic modulus 700-800 MPa, bending elastic modulus 470-850 MPa, specific impact strength without notching > 100 kD/m^2, HB hardness 30-40 MPa, Vick's heat resistance 373-383 K, brittleness temperature < 4 K, glass transition temperature 153 K, melting point 590 K, decomposition temperature > 688 K.

Polytetrafluoroethylene is also undergoing structural modification [11]. The polymer passes mechanical activation, shock wave and electromagnetic treatment, and radiation exposure.

For the mechanical grinding of dry PTFE powder, the MRP-1M crusher was used with the following parameters: sample load: 120 ± 20 g, productivity - 0.6 kg/h, chamber volume - 0.4 m^3, knife diameter - 0.345 m, maximum linear knife speed Vmax = 130 m/s. The number of rotations of the knife (working body) was: n = 5000 min^{-1}, n = 7000 min^{-1}, n = 9000 min^{-1}, crushing time: 3 min, 5 min, 8 min. It was determined that the optimal rotation frequency is equal to n = 9000 min^{-1} and the time interval is equal to τ = 5 min (Table 1), at which the maximum indicators of the physicomechanical and tribotechnical properties of the polymer are obtained.

As a result of mechanical activation, the tensile strength increases by 2.6 times, the relative elongation at break by 4.3 times, and the wear rate decreases by 1.85 times compared to non-activated PTFE [12].

II. Chemical, thermal and mechanical activation of the filler

It is known that the introduction of carbon fibers as fillers affects the tribotechnical properties of polytetrafluoroethylene, which was studied in [13]. It was found that the wear resistance is higher compared to that of pure PTFE.

The carbonized carbon fibers obtained from organic hydrate cellulose (HC) tissue were studied. The carbon fiber dispersions were pretreated with a mixture of flame retardants $Na_2B_4O_7 \cdot 10\ H_2O$ and $(NH_4)_2HPO_4$, taken in equal amounts. Next, the carbon fiber was heat-treated in an environment of natural gas CH_4 by passing through a Tammany Furnace and dry nitrogen N_2, gradually increasing the temperature from 723 K to 2673 K, and the types of fibers were obtained, which are given in (Table 2), where HC is hydrate cellulose, Tc is the final temperature of carbonization or graphitization, LM is low-modulus carbon fiber. The classification of fibers is given in [14].

Thus, from the initial hydrate cellulose fabric at a temperature of 723 K in methane (CH_4), a partially carbonized UT-4 fabric with the following characteristics was obtained (Table 3). By further annealing the UT-4 fabric at 1123 K in a CH_4 atmosphere, a carbonized UTM-8 fabric with the following characteristics was obtained (Table 3). The UTM-8 fabric was heat-treated at 2673 K in an N_2 atmosphere and a graphitized TGN-2m fabric was obtained. The resulting fabric had the following characteristics (Table 3). After the heat treatment, the carbon fiber fabric TGN-2m contained the most carbon (99.2 %) compared to UT-4 and UTM-8.

After grinding in MRP-1, the dispersions of UTM-8 carbon fibers were treated with a solution of $Na_2B_4O_7 \cdot 10H_2O + (NH_4)_2HPO_4$ in a 1:1 ratio and heat-treated sequentially at temperatures 1123 K, 1473 K, 1623 K, 1873 K, 2273 K, 2573 K, 2673 K in a dry nitrogen environment and carbon fibers TGN-T850, TGN-T1200, TGN-T1350, TGN-T1600, TGN-T2000, TGN-T2300, TGN-T2400 were obtained, accordingly. Graphitized TMP-3 fibers were obtained from TGN-2m by applying a pyrocarbon coating in N_2 at 2673 K. However, filling with such a fiber leads to a decrease in the antifriction

Table 1.

The effect of mechanical activation on PTFE properties

Modes of mechanical activation	Tensile strength,, σ_{pp}, MPa	Relative elongation at break δ, %	Wear rate I·10^{-6}, mm^3/N·m
Not activated	9.5	96	1133
τ = 3 min, n = 5000 rpm	10.2	240	1080
τ = 5 min, n = 5000 rpm	21.6	416	930
τ = 8 min, n = 5000 rpm	17.3	280	800
τ = 3 min, n = 7000 rpm	10.7	270	970
τ = 5 min, n = 7000 rpm	23.5	423	820
τ = 8 min, n = 7000 rpm	18.2	358	717
τ = 3 min, n = 9000 rpm	19.6	290	890
τ = 5 min, n = 9000 rpm	24.8	415	610
τ = 8 min, n = 9000 rpm	18.0	340	720

Table 2.

Carbon fibers

Mark	Source organic fiber	Type of carbon fiber	Tк, K
UT-4	HC	LM	723
UTM-8	HC	LM	1123
TGN-2m	HC	LM	2673
TMP-3 (Pc: 12% pyrocarbon)	HC	LM	2673
TKK-1 (Pk: 0,2-3,0 % SiC)	HC	LM	2673
TKC-1 (Pk: 0.8-4.0 % ZrC)	HC	LM	2673
TGN-T850	HC	LM	1123
TGN-T1200	HC	LM	1473
TGN-T1350	HC	LM	1623
TGN-T1600	HC	LM	1873
TGN-T2000	HC	LM	2273
TGN-T2300	HC	LM	2573
TGN-T2400	HC	LM	2673

Table 3.

Characteristics of carbon fibers at the stage of their production [6].

Fiber characteristics	UT-4	UTM-8	TGN-2m
tensile load of the fabric (on the warp), N/cm	100-160	70-240	150-160
breaking load of the fabric (by weft), N/cm	15-30	20-100	25-35
Carbon content, %.	60-70	66-72	99.1-99.3
Hydrogen content, %.	-	-	0.2-0.4
Boron compounds content, %.	0.2	3.0-3.6	0.4
Phosphorus content, %.	0.5	3.0-3.6	0.002
Sols content, %	1.5	21-26	0.45-0.55
Fiber strength at break, GPa	0.2-0.3	0.5-0.6	0.45-0.50
Tensile modulus of elasticity, GPa	3-4	30-50	30-50
Relative elongation at break, %.	4.5-6.5	4.5	1.2-1.3
Fiber diameter, μm	11-14	10-12	8-9
Thermal conductivity, W/(m·K)	0.08-0.09	0.08-0.12	0.14-0.16

properties of composites [15].

To fill the composite, carbon fiber material (CFM) in the form of fabric and fiber was introduced into the matrix material at a certain temperature regime (T_k=723-3073 K) [16].

Mechanical activation. Previously, the CFM in the form of fabric or ribbon was crushed in a crusher KDU-2.0 "Ukrainka" (number of crushing hammers - 90, number of knives - 3, number of rotations of working bodies n = 1440 rpm, capacity - 200 kg/h). The obtained fiber fractions were 3.0-15.0 or 0.5-8.0 mm. [17].

According to the CMA technology, the carbon fibers were mainly crushed in the MRP-1 (knife diameter - 0.205 m, maximum linear knife speed V_{max} = 78 m/s, knife rotation frequency f = 120 s^{-1} (117 s^{-1}), knife rotation number n = 7000 rpm).

Mechanical crushing of the fiber was also carried out in a crusher MRP-1M (sample load: 120 ± 20 g, productivity - 0.6 kg/h, knife speed n = 7350 rpm, chamber volume - 0.4 m^3, knife diameter - 0.345 m, maximum linear knife speed V_{max} = 130 m/s). The MRP-1M was also used as a composition mixer.

The CMA technology is based on high knife speeds, which crush both the fiber and the composition; the tests were carried out from 2000 to 12000 rpm.

The carbon fibers pre-ground in MRP-1 or MRP-1M (to a bulk density of 40-160 kg/m^3) were subjected to fine grinding in a ball mill. For grinding, the carbon fiber dispersion was loaded into the ball mill chamber, the working part of which had a volume of 92.66 cm^3 of steel, a diameter of 65.5 mm, and a height of 27.5 mm. Thirty-five balls of steel SCH-15 with a diameter of 12.7 mm were placed in the mill chamber (filling the working part of the mill with 48 volume %). The number of mill rotations ranged from 20-30 to 125-175 per minute. The tests were carried out for 5-2050 hours in a humid air environment.

The carbon fibers were also subjected to intensive mechanical grinding in a D-160 Z crusher from Alpine (capacity - 20-40 kg/h, rotor diameter 0.160 m, number of pins (fingers) on the rotor disk - 316). RPM range: 3000 rpm, 5000 rpm, 7000 rpm, 9000 rpm, 11000 rpm,

14000 rpm, 17000 rpm, 19000 rpm, 22500 rpm. The length of the carbon fiber is $\ell_0 \leq 5$ mm, ℓ_τ = 40-150 μm. [18]. However, it was found that the grinding of carbon fiber in a dismemberer is not effective.

The technology and final heat treatment temperature have an impact on pH. For example, for partially carbonized fibers, the pH is in the range of 4.8-6.4, for carbonized fibers 6.4-7.8, and for graphitized fibers 7.3-8.3. The pH is also affected by the time of fiber crushing. As the crushing time increases, the pH decreases.

During the heat treatment of fibers, the flame retardants applied to the fibers decompose (above 373 K) according to the scheme:

$$(NH_4)_2HPO_4 \rightarrow H_3PO_4 + 2NH_3\uparrow$$

With a gradual increase in temperature, H_3PO_4 loses water and polymers of the composition $(HPO_3)_n$ are formed.

Filling composites with low modulus carbon fibers increases the isotropic roughness of the contact metal surfaces by $\sqrt{m_0}$ = 0,043 – 0,143μm [19].

The activation of carbon fibers by zirconium dioxide ZrO_2 + 3 % Y_2O_3 at 700^0C significantly affects the intensity of volumetric wear of fluoroplastic carbon fiber reinforced plastic [20].

III. Mixing of components and technology of composite materials production

For mixing polymer powders and fibers, a crusher with high-speed submersible working knives MRP-1 with n = 7000 rpm was used. To do this, in one case, long fibers 0.6-6 mm long were added to the polymer composition, which was crushed in a crusher KDU-2.0 "Ukrainka". And in the second case, the fibers, after being crushed in the KDU-2.0 "Ukrainka" for 3-30 minutes before mixing the polymer composites, were crushed in the MRP-1 to lengths of 20-500 μm, and then mixed with PTFE powder and molybdenum disulfide in the MRP-1 for 5 minutes.

This mixing technology results in not only mechanical mixing. Due to the additional grinding of the polymer composition components, triboactivation of the mixture particles occurs [21].

The resulting slurry was subjected to cold pressing under a pressure of Pp = 50.0-70.0 MPa to obtain blanks. The next process was sintering the blanks in air at 638 K. The heating-cooling rate was 40 K/h.

The resulting material (GOST 12015-66, GOST 12019-66) was left at room temperature for 15 days for testing. After aging, the material was conditioned for 24 hours.

IV. Research of physical and mechanical properties

The physico-mechanical properties of the composite material based on polytetrafluoroethylene are influenced by the parameters of carbon fiber distribution [22], technological parameters [23], the content of the components of the composite material [24, 25].

A study of the influence of technological parameters on the physical and mechanical properties of composite samples is given in [26]. Using the methods of mathematical planning of the experiment, it was found that the intensity of wear is most affected by the mixing time of the composition. The content of low-modulus carbon fiber, the time of its preliminary grinding, and the mixing time also affect the friction coefficient, tensile strength, impact strength, and mechanical properties. The content of solid lubricant in the composition affects the tensile strength.

It has been found that the type [27] and shape of carbon fiber [28], composition and degree of preliminary deformation [29] affects the wear resistance and thermal conductivity and, accordingly, the thermal properties of PTFE composites. The orientation of the fibers also affects the thermophysical properties [30,31].

The concentration of fillers in the composite has a significant impact on the wear rate. With an increase in the content of carbon fiber in the polymer matrix, an increase in wear resistance is observed, but, having reached a certain optimal concentration, it begins to decrease due to the gradual destruction of components [32]. Amorphization processes occur when the concentration of carbon fiber is in the range of 3-4 %, the maximum wear resistance is achieved at a content of 15 % [33].

The study of physical and mathematical properties by UV spectroscopy [12] showed an increased concentration of CF_2 groups in PTFE after mechanical activation. The tests were performed on a Specord 75 IR spectrophotometer. An increase in the speed during the activation of PTFE contributes to an increase in the end groups of CF_2 in its composition.

The pH-metric analysis of the aqueous extracts of carbon fibers UT-4 (T_k = 723 K), UTM-8 (T_k = 1123 K), and TGN-2m (T_k = 2673 K) is given in [8]. The studies have shown that the pH change is most affected by the time of crushing and the type of fiber. The pH of the aqueous extracts of the initial carbon fibers have the following values (Fig. 1). After additional grinding for 5 minutes to a bulk density of 80 kg/m^3 in MRP-1, the pH of the aqueous extracts of carbon fibers decreases relative to the original fibers by 0.3, 0.43, 0.50, respectively, for UT-4, UTM-8, and TGN-2m. Thus, any thermomechanical activation of carbon fiber leads to a decrease in the pH of aqueous fiber extracts.

Work [34] found a linear relationship between the wear resistance and friction coefficient of PTFE-based carbon fiber reinforced plastics filled with short, randomly arranged carbon fibers and the mechanical and thermophysical properties of adjacent surfaces. Wear intensity studies were conducted at the HTI-72 installation [18]. The analysis of the test results shows that wear resistance does not depend on the hardness of adjacent friction surfaces, but is significantly affected by the plastic deformation energy of the adjacent surface of the counterbody and the tensile deformation energy.

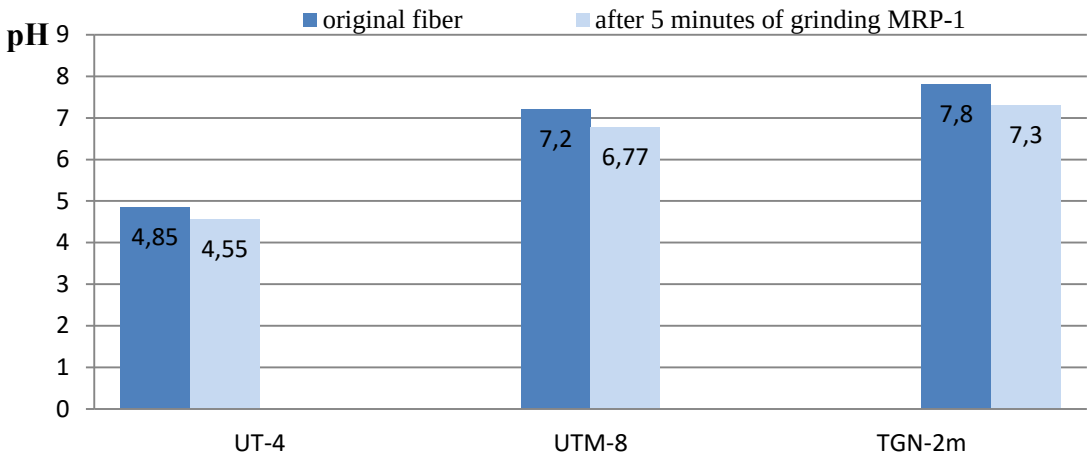

Fig. 1. pH of water extracts from the initial carbon fibers (exposure for 15 min. at 293 K, tub modulus 33) and after 5 min. grinding in MRP-1.

The thermographic analysis. Carbon fibers with a final heat treatment temperature of 1123, 1473, 1623, 1873, 2273, 2573 K, which were previously crushed in a crusher to a bulk density of 0.04 g/cm^3, were studied. These fibers were annealed in the temperature range of 293-953 K. Additionally, the fibers were ground for 200 hours in a ball mill and also annealed at 293-953 K. The results of the thermographic analysis are given in Table 4 and Table 5.

After annealing the initial fiber, the temperatures of the exo-effect onset T_{0max} (Table 4) and peak T_{max} (Table 5) shift to the high-temperature region. After an additional 200 hours of dispersion of the original carbon fiber, these indicators shift to the low-temperature region, but after annealing the chopped fibers, there is a shift of these indicators to the high-temperature region. Probably, the complexes formed on the surface of carbon fibers are converted into gas as a result of thermal effects.

Table 4.

The temperature of the onset of the exo-effect (T_{0max}) of the studied carbon fibers TGN-T

Final heat treatment temperature of carbon fiber, K	T_{0max}			
	Before annealing		After annealing	
	Starting point	After 200 h of grinding	Starting point	After 200 h of grinding
1123	406	386	520	510
1473	543	390	640	540
1623	582	396	662	545
1873	610	400	680	550
2273	645	405	700	600
2573	675	410	729	610

Table 5.

The temperature of the peak exo-effect (T_{max}) of the studied carbon fibers of TGN-T

Final heat treatment temperature of carbon fiber, K	T_{max}			
	Before annealing		After annealing	
	Starting point	After 200 h of grinding	Starting point	After 200 h of grinding
1123	620	542	757	713
1473	798	584	795	728
1623	825	605	825	735
1873	870	615	860	742
2273	876	635	878	760
2573	880	650	903	770

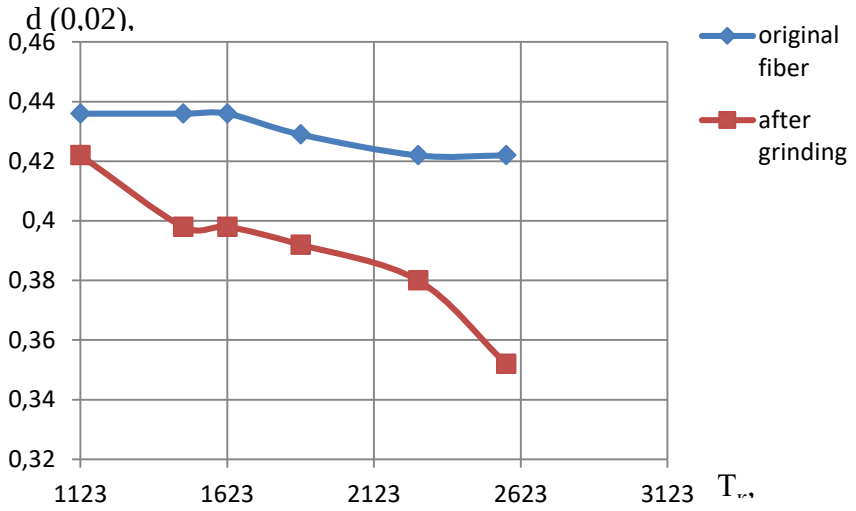

Fig. 2. Dependence of the interlayer distance on the final heat treatment temperature of the initial fibr of TGN-T and the fiber after 200 h of grinding in a ball mil.

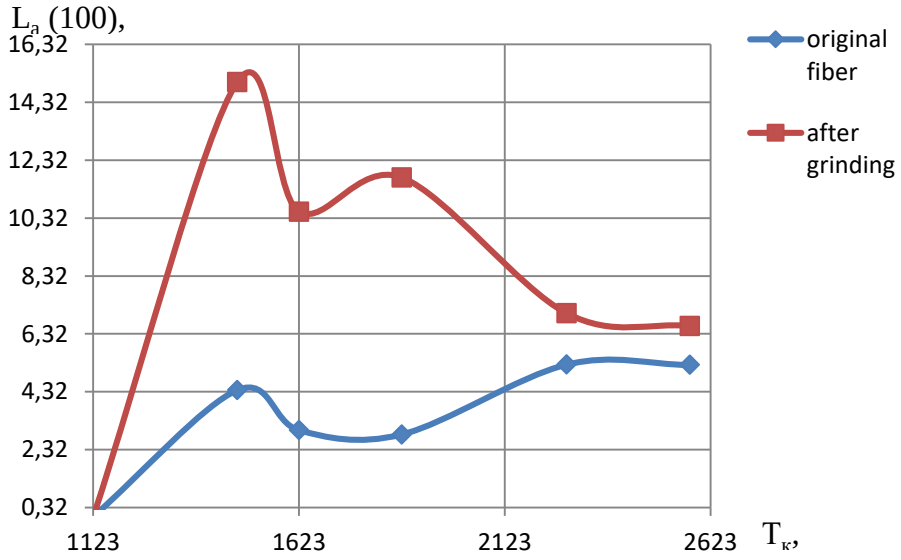

Fig. 3. Dependence of the thickness of the hexagonal layer package on the final heat treatment temperature of carbon fibers TGN-T.

Mechanical grinding and the temperature of the final heat treatment (T_k) of carbon fibers lead to the improvement of the turbostrata structure. Fig.2 shows that with an increase in the final heat treatment temperature of TGN-T carbon fibers from 1123 to 2673 K, the interlayer distance d (002) decreases for the original fiber and significantly decreases after 200 hours of mechanical activation in a ball mill. The length of the package of layers of L_a(100) hexagons (Fig.3) increases significantly after 200 h of mechanical dispersion but decreases with increasing T_k. Therefore, thermomechanical activation contributes to the formation of an ordered two-dimensional structure of carbon fibers and increases the wear resistance of the polymer composite.

Conclusions

It has been summarized the impact of CMA technology on the properties of composite materials, which are obtained by the authors and many researchers in this scientific area.

The complex application of chemical treatment, mechanical grinding and activation of fillers and matrix have a positive effect on the mechanical and tribotechnical properties of the polymer composite.

Composite materials obtained by CMA technology have higher strength and wear resistance than materials produced using traditional technology.

It was revealed that chemo-mechanical activation increases the thermal stability of composites based on polytetrafluoroethylene, and also affects its

supramolecular structure, changing from disordered to more organized.

It is determined that the mechanical activation of carbon fibers and PTFE polymer matrix increases by 10-25 % the physico-mechanical properties of the composition as a whole, which has a further effect on friction and wear.

A comparative analysis of tribotechnical parameters, tensile strength characteristics before and after chemo-mechanical activation was carried out.

The optimal temperature conditions of operation were found.

Thus, CMA technology is effective and can be used for industrial realization.

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Особливості хемо-механо-активаційної технології одержання полімерних композитних матеріалів

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Проведено огляд робіт по дослідженню процесів одержання полімерних композитів за хемо-механо-активаційною технологією (ХМА-технологією). Зроблено аналіз та узагальнення методів дослідження і випробування композиційних матеріалів на основі політетрафторетилену (ПТФЕ) і вуглецевих волокон. Встановлено вплив ХМА- технології на структуру і властивості ПТФЕ-композиту.

Ключові слова: полімерний композит, політетрафторетилен, вуглецеві волокна, хемо-механо-активаційна технологія.