

PHYSICOCHEMICAL PRINCIPLES OF THE TECHNOLOGY OF FORMATION OF POLYMER COMPOSITE MATERIALS BASED ON POLYTETRAFLUOROETHYLENE — A REVIEW

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The experience of many theoretical and practical works concerning the technology of PTFE composite formation has been analyzed and systematized. The characteristic feature of the work is that PTFE composites reinforced by carbon fibers are considered to be complex objects with the properties characteristic of tribotechnical material science of composite materials. The structure complexity of such objects provides their consumer properties due to many factors that influence the significant increase of physicomachanical characteristics. The research results defined the operation regimes of process equipment: after the processing the PTFE composites have increased operation properties in comparison with the basic ones and can be most effectively used for industrial implementation.

KEY WORDS: *polymer composites, processing technologies, adhesion, material testing, molding, thermal vacuum technology, thermal treatment, mechanical properties*

1. INTRODUCTION

The modern industry requires the production of new polymeric and composite materials (PCM) with high performance characteristics (Jose et al., 2012; Ivashchenko et al., 2014; Pogrebnyak 2013a,b; Pogrebnyak et al., 2007, 2001). The strategic way of creating such materials is to use different approaches to modifying the known materials (Asadi et al., 2016; Vail et al., 2011; Vas et al., 2007; Bijwe and Sharma, 2013; Park and Shin, 2016). A very important issue that defines the technical and economic efficiency of the use of new composite materials is the forecast of operating properties and modeling of PCM related to this problem. Therefore, the further development of the production of PCM on the basis of polytetrafluoroethylene (PTFE) should be based on the scientific principles of the technology of polymers and composites, applied materials science, and physicochemical mechanics of composite structures (Smith et al., 2014; Drobny 2009; Misaelides et al., 2004; Lavrentiev and Pogrebnyak, 1998; Maksakova et al., 2015; Pogrebnyak et al., 1987, 2015; Kumar et al., 2010).

Carbon fiber-reinforced plastic (CFRP) on the basis of polytetrafluoroethylene and modified fillers that have successfully replaced traditional tribotechnical materials are the advanced materials to be used in friction units of industrial equipment (Park and Seo, 2012; Venkateswarlu et al., 2014; Masuelli, 2013; Khedkar et al., 2002).

The grounded operating conditions of the matrix and fillers and PTFE composite manufacture affect the physicomechanical and tribotechnical properties of CFRP.

For a long time the development of PTFE-based PCM technology was founded on empirical research. This is understandable if we take into consideration the complexity of interfacial interactions in multicomponent polymer systems and their connection with the properties of polymer composites. The combination of theoretical research of structural and phase transformations occurring on filling the PTFE matrix of a composite with fillers and experimental research is the way allowing one to formulate a scientifically based approach to forecasting and targeted regulation of the PTFE composite properties.

Currently, scientific substantiation of the choice of crushing equipment for making polymer composite systems (using modern methods of physical and mathematical modeling) and efficient modes of its operation either do not exist, or they are narrowly scaled (industry). The questions concerning the technology of forming (pressing) the composition of a composite material, which leads to obtaining preparations with various densities and, consequently, the heterogeneity of the properties over the volume of a preparation, are not fully resolved. The question of thermal treatment of formed preparations have not been studied theoretically at all and therefore one mode of thermoformation is practically realized that quite often leads to emergence of defective preparations at this stage of technological process. The process of ther-

momechanical effect on the structure and properties of a polymer composite still has not been considered at all as yet and therefore requires both fundamental and practical investigations.

Identifying the influencing pattern of fillers, chemicotechnological factors on the basis of deepening scientific understanding of the structure formation processes of composites with hybrid fillers, studying their physicommechanical and tribotechnical characteristics allow one to control the properties of PTFE composites, which is one of the important problems of modern polymer materials science. The study of the matrix and preparation of fillers and development of the PTFE-based composites solves an important scientific and technical problem and determines the relevance of this paper.

The authors have started to work on some aspects of this problem under the guidance of Ph.D., associate professor Anatoliy F. Budnik (13.08.1951–20.12.2015) and they are continuing this work now. A considerable number of research and commercial works carried out allow stating the experimentally proven concept that interfacial and surface phenomena of polymer–solid and composition methods play a decisive role in the imparting of the PTFE composites properties.

The aim of the paper is to generalize the results of research of the influence exerted by the process of technological preparation of components on the physical, mechanical, and performance properties of composite materials and determine their optimum values (rotational speed of hog shredder working parts, milling period, modes and mixing period of the composition ingredients, parameters and modes of the pressing composition in half-finished products, modes and monolithization (sintering) period of half-finished products, thermomechanical effect modes on the properties of the resulting composite).

The results of the science-based solution of such problems provides a possibility of creating a technology of PTFE composite materials and furnishes consumers with composite materials with predicted properties up to the best world analogs.

2. OBJECTS AND METHODS OF RESEARCH

2.1 Research Objects

Polytetrafluoroethylene is a high-molecular composition whose molecules consist of a large number of identical atomic groups ($-\text{CF}_2-$) with: $(-\text{CF}_2-\text{CF}_2-)_n$ chemical bonds. Tetrafluoroethylene is a primary monomer for PTFE. The resulting polymer is a loose, fibrous, and cloggy white powder (Venkateswarlu et al., 2014; Biswas and Vijayan, 1992).

Depending on its technical application, PTFE is produced in several grades, such as F-4, F-4PN-90, F-4PN-40, F-20 4PN, etc. (GOST 10007).

It was found experimentally that PTFE F-4 used for these studies is the most relevant to the requirements set (Table 1).

PTFE is one of amorphous-crystalline polymers with the melting point of the crystallites $+327^{\circ}\text{C}$ and a vitrification temperature of the amorphous phase -120°C ; it has a high degree of crystallinity; the large number of crystallites is observed even after quenching (rapid cooling starting from the melting point) (Drobny, 2009; Biswas and Vijayan, 1992).

The mechanical properties of PTFE are considerably determined by the crystallinity degree, i.e., the content of the crystal phase in the structure of a polymer (Biswas and Vijayan, 1992). The degree of crystallinity of PTFE depends on the speed of cooling in the case of thermal treatment (sintering) of pressed materials in the range of temperatures $370\text{--}300^{\circ}\text{C}$. The maximum content of the crystal phase is observed at a minimum speed of cooling when favorable conditions for forming crystallites are created (Drobny, 2009).

The material has an abnormally low friction coefficient (0.01–0.04), but it is disposed in cold flow which can be lowered by the introduction of fillers.

Hydrated cellulose carbon fiber (CF) UTM-8-1s (Technical Specifications 48-20-17-77) obtained by chemical processing in an aqueous solution of fire-retardants $\text{Na}_2\text{B}_4\text{O}_7 \cdot 10\text{H}_2\text{O} + (\text{NH}_4)_2\text{HPO}_4$ and annealed at the temperatures $723 \pm 20 \text{ K}$ in the natural gas CH_4 environment was used as the main fiberfill.

Its chemical composition and properties are listed in Tables 2 and 3.

2.2 Methods of Research

Test samples were obtained by the cold molding technology (molding pressure $P_{\text{mold}} = 50.0\text{--}70.0 \text{ MPa}$), followed by free sintering of tablet blanks in air at $365 \pm 5^{\circ}\text{C}$ at a heating–cooling rate of 40°C/h .

TABLE 1: Characteristics of brand F-4

Appearance	White powder, which easily lumps without visible impurities
Mass fraction of moisture, %, not more	0.02
Density, g/cm^3 , not more	2.18–2.21
Breaking strength nonhardened sample, MPa (kgf/cm^2), not less than	15 (150)–26 (260)
Relative elongation at break of nonhardened sample, %, not less than	150–350
Dielectric strength, kV/mm , not less	50–60
Average particle size, μm	100–180 (is not normalized)

TABLE 2: Chemical composition of UTM-8-1s carbon fabric

C, %	H, %	O, %	B, %	P, %	Ash, %
60–65	1.1–4.5	3.5–4.5	3.0–3.6	3.0–3.6	21–26

TABLE 3: Mechanical properties of UTM-8-1s carbon fabric*

Density ρ , g/cm ³	Breaking strength in warp, N/cm	Breaking strength in transverse direction, N/cm	Breaking strength, GPa	The elastic modulus, GPa	Coefficient of thermal conduc- tivity, W/(m·K)
1.510	70–235	20–100	0.52–0.60	27–47	0.08–0.12

*The diameter of the fibers is 10–12 μm .

The methodology of studying the composite properties included determination of the density ρ (g/cm³), breaking strength σ_b (MPa), relative elongation δ (%), and wear intensity $I \cdot 10^{-7}$ mm³/N·m in accordance with the regulations.

Tests of strength and relative elongation at break were performed on ring samples of 50 × 40 diameter and 10 mm height using rigid half-discs (GOST 11262) at R-1 disruptive installation (GOST 4651) at a speed of motion of the sliding member of 0.25 cm/min.

The density ρ (g/cm³) of the samples was determined by hydrostatic weighing (GOST 15139).

The study of the wear rate was carried out on an SMT-1 serial friction machine according to the "partial insertion-shaft" scheme.

A set of samples were tested in a friction mode without external lubrication at a sliding speed of $V = 1$ m/s and pressure of $P = 1$ MPa. The counterbody was a roller $\varnothing 48$ mm of steel 45 (HRC 25, Ra – 0.38 μm). Partial insertion was manufactured of PTFE and was a sector of width 16 mm from the ring $\varnothing 80$ to $\varnothing 60$ mm and of height of 9 mm.

The rate of mass wear was assessed by mass loss of samples per unit time. The magnitude of wear of the samples was determined gravimetrically on an analytical balance within the accuracy of 10^{-5} g and transferred to the intensity of wear. The moment of friction was recorded using a Termodat 17E3 device.

In assessing the intensity of wear, the mean square error was regulated by errors of measurement of mass of a sample, speed and duration of friction and they did not exceed 5%.

The study of the supramolecular structure of activated PTFE powder was carried out using a TESCAN MIRA 3 LMU scanning electron microscope of high resolution.

The surface morphology of CF was carried out using an REM-200 raster electron microscope.

Experimental data were processed by the methods of mathematical design of experiments and mathematical statistics.

3. METHODS OF PTFE MATRIX MODIFICATION

Despite the unique characteristics of PTFE, it has a number of properties that limit its use as a tribotechnical material. These include (Khedkar et al., 2002):

- high tensile creep loading that appears at 3 MPa of tensile loading at normal temperatures;
- high thermal expansion coefficient at normal temperatures that changes abnormally at a temperature in the range of structural phase transitions (temperature range from 280 to 310 K);
- low thermal conductivity (10–50 times less than in metals);
- low wear resistance under dry friction conditions especially at a high sliding velocity.

The heat released in the friction zone due to the low thermal conductivity facilitates the significant rise of temperature on the friction surface. As a result, the physical and chemical processes that reduce the strength and stiffness of the polymer are intensified and the linear sizes are greatly increased, which can eventually lead to destruction of the friction unit. Thereby it is necessary to use physical and chemical methods of PTFE modification.

A review of the literature and patents (Sirenko, 1985; Pugachev and Roslyakov 1987; Technical Specifications, 1989; Lipatov, 1977) has revealed the low potential of traditional technological approaches in obtaining PTFE-based PCM. However, various advanced technological methods that allow obtaining composite materials and products with the required performance properties may be implemented. They are the synthesis of polymer matrices of different compositions and structures (Ruiz et al., 2011; Buznik et al., 2013); composition of a polymer and oligomer mixture with different levels of interaction (Utracki and Wilkie, 2014); modification of matrices of various origins by aimed restructuring and structural interaction energy impact; matrix modification by additions of activators of different sizes, shapes, and properties (Mikhailova et al., 2011; Budnik et al., 2015a); formation of nanophase matrices with significantly different characteristics (Okhlopko et al., 2008).

It should be noted that the industrial implementation of these technologies is associated with significant energy, material, and labor expenses and requires the managerial study of the price–quality relationship.

The main modification methods are shown in Fig. 1.

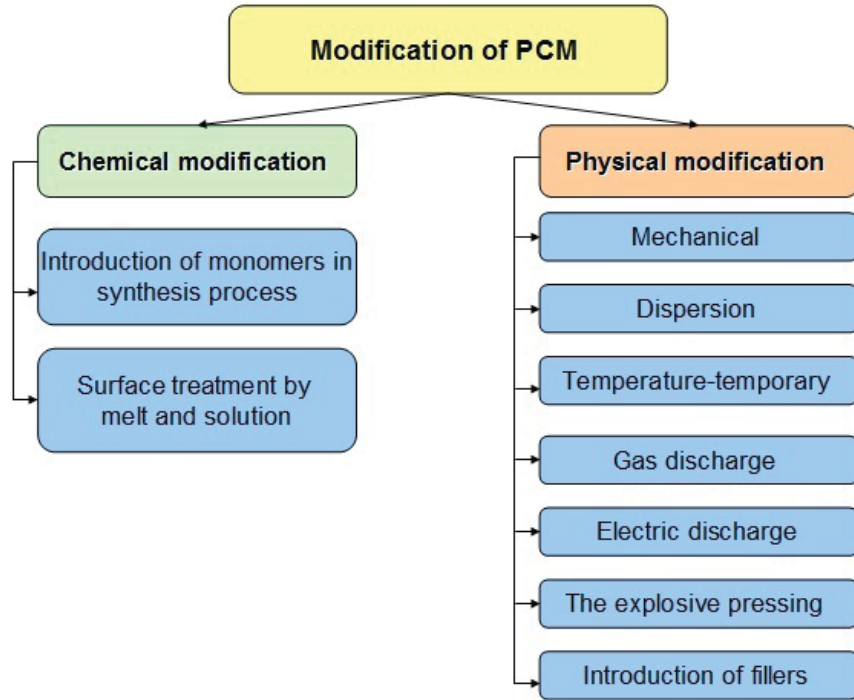


FIG. 1: Scheme of the main methods of PCM modification

4. INDUSTRIAL TECHNOLOGY FOR PCM WITH A PTFE MATRIX

The technology of filled polymers, first of all, is the process of mixing initial components. It determines the ultimate macro- and microstructure of composite materials, their physicomechanical and tribotechnical characteristics. The physicomechanical and tribotechnical characteristics of filled polymers relate not only to interaction between components, but also to the change in the structure and properties of PCM associated with phase and structural transformations.

The technological process of obtaining composite PTFE-based materials had undergone a change on the difficult way of its development (Pugachev and Roslyakov, 1987).

At present, the industrial process of obtaining composites based on PTFE with CF (Technical Specifications, 1989) consists mainly of the following chain of operations (Fig. 2): during the process of sintering of pressed material at the stage of fusion of PTFE particles and fillers, the chemical, physical, and mechanical performance properties of the future product are laid, so the sintering and the subsequent heat treatment are an important step in the process of manufacturing products from a composite.

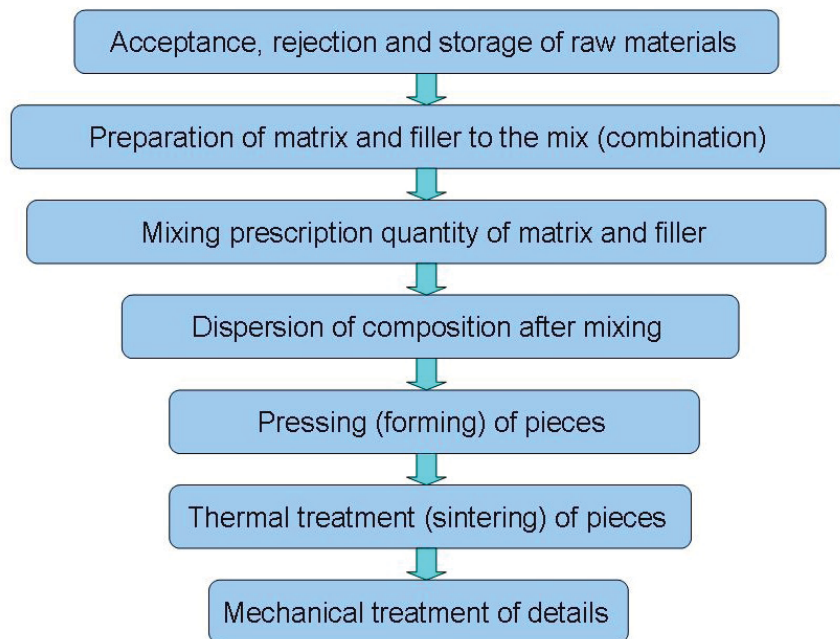


FIG. 2: Scheme of the industrial process of obtaining composites based on PTFE

In the practical application it is not always possible to achieve the best production modes of obtaining PTFE composites. The solution is seen in the maximum intensification of each stage of the process.

The structural links of this chain may be supplemented by new elements (processes) or vice versa, some parts of the links may be excluded from the process as unnecessary. The comprehensive approach to each stage of the PTFE composite making will identify the most important factors of energy fields impact on the structure-forming activity of the composition ingredients and identification of the criteria of their compatibility. At the same time, the technological modes of gradual impact on the composition (drying, milling, mixing, pressing, and heat treatment) play an important role as well as the ordering of structure elements of the composition and activation of physical and chemical interaction at the phase boundary.

5. PREPARATION OF PTFE MATRIX BY MECHANICAL ACTIVATION

Activation of PTFE powder was carried out by an MRP-1M mill with varying frequency of working parts rotation in the interval $n = 5000\text{--}9000 \text{ min}^{-1}$ and during experimentally determined time interval $\tau = 3\text{--}8 \text{ min}$ (Budnik et al., 2015b).

The morphology and fractional composition of PTFE on mechanical activation with a number of rotations less than 5000 min^{-1} do not provide equal distribution of activated PTFE particles in the material, and with a number of rotations more than 9000 min^{-1} it leads to coagulation of activated particles and formation of a heterogeneous material structure.

The activated powder was obtained by dry milling in an MRP-1M high-speed blade mixer. The difference between the structure of activated and nonactivated PTFE leads to differences in the physical and mechanical properties (Table 4).

Mechanochemical destruction of PTFE macromolecules occurs in previous activation with the formation of radical fragments. The presence of the active surface of the filler particle, on the one hand, and PTFE macromolecule free radical, on the other hand, may initiate the polymer grafting reaction to the filler. Although the reactions of chemical bonds formation between the polymer and the filler surface take place only in active centers and have a likely nature, their contribution to strengthening of the composite material is very significant.

This also leads to the preservation of extra energy by the polymer substance, to the changes of the thermodynamic properties, and to the increase in its reactivity.

Furthermore, the mechanical load, as a result of the particles collisions, leads to metastable states of surface layers of polymer particles. These particle collisions progress for several seconds, and are followed by the local increase in the temperature and pressure at the contact surfaces points. All these phenomena lead to the formation of uncompensated valences on the surface, promote the interaction between the filler particles of the composite, and initiate the polymerization reaction of monomers or formation of chemical bonds with polymer radicals.

TABLE 4: Effect of PTFE mechanical activation on physical, mechanical, and tribotechnical properties

No. of sample	Technology of obtaining	Density ρ , g/cm^3	Breaking strength σ_b , MPa	Relative elongation δ , %	Wear intensity $I \cdot 10^{-7}$, $\text{mm}^3/\text{N} \cdot \text{m}$
1	nonactivated	2.269	9.5	96	113.3
2	$\tau = 3 \text{ min}$, $n = 5000 \text{ min}^{-1}$	2.208	10.2	240	108.0
3	$\tau = 5 \text{ min}$, $n = 5000 \text{ min}^{-1}$	2.211	21.6	416	93.0
4	$\tau = 8 \text{ min}$, $n = 5000 \text{ min}^{-1}$	2.175	17.3	280	80.0
5	$\tau = 3 \text{ min}$, $n = 7000 \text{ min}^{-1}$	2.199	10.7	270	97.0
6	$\tau = 5 \text{ min}$, $n = 7000 \text{ min}^{-1}$	2.205	23.5	423	82.0
7	$\tau = 8 \text{ min}$, $n = 7000 \text{ min}^{-1}$	2.211	18.2	358	71.7
8	$\tau = 3 \text{ min}$, $n = 9000 \text{ min}^{-1}$	2.203	19.6	290	89.0
9	$\tau = 5 \text{ min}$, $n = 9000 \text{ min}^{-1}$	2.214	24.8	415	61.0
10	$\tau = 8 \text{ min}$, $n = 9000 \text{ min}^{-1}$	2.213	18.0	340	72.0

It is determined that the best achieved result is the mechanical activation mode of PTFE matrix with the rotation number of mill working parts $n = 9000 \text{ min}^{-1}$ for 5 min. In addition to the above, breaking strength $\sigma_b = 24.8 \text{ MPa}$, relative elongation $\delta = 415\%$, and the wear intensity $I = 61.0 \cdot 10^{-7} \text{ mm}^3/\text{N}\cdot\text{m}$. Nonactivated PTFE has $\sigma_b = 9.5 \text{ MPa}$, $\delta = 96\%$, and $I = 11.33 \cdot 10^{-7} \text{ mm}^3/\text{N}\cdot\text{m}$.

The energy impact on unfilled PTFE increases the parameters of its deformation and strength characteristics (breaking strength by 2.6 times, breaking elongation by 4.3 times) while maintaining the high tribotechnical indices. This is obviously due to the formation of new reaction centers and increase in the individual fragments of macromolecules surface energy as a result of the elastic and plastic deformations.

Increasing the PTFE wear resistance in the mechanical activation is associated with the decrease in the crystalline degree and the increase in the average interlayer distance during the frictional interaction and the structural adjustability of modified PTFE under conditions of friction and demonstration of synergistic effects of self-organization tribostructures with a high wear resistance.

Using the methods of electron microscopy, it has been established that the PTFE supramolecular structure under mechanical activation significantly changed from a disordered lamellar structure to a spherulitic structure with higher ordering (Fig. 3).

In the structure of PTFE samples after mechanical activation, strands of fibers with length from 10 to 50 μm and diameter from 10 to 100 nm (Fig. 3b) and "frost flowers" (Fig. 3c) are observed which are absent in a nonactivated PTFE structure (Fig. 3a).

The difference between the particle morphology of factions is due to the fact that the products obtained at different thermobaric effects have a different ratio of molecu-

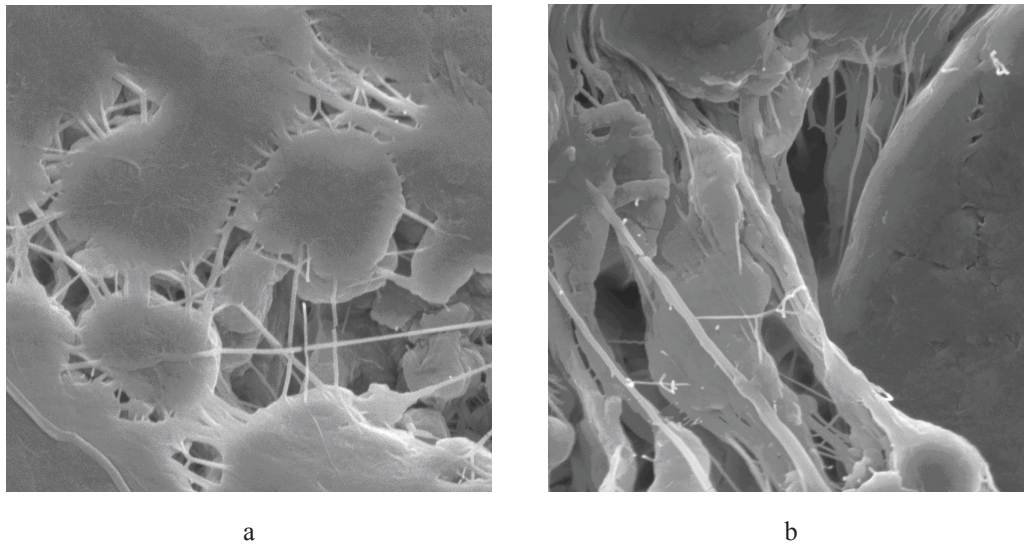
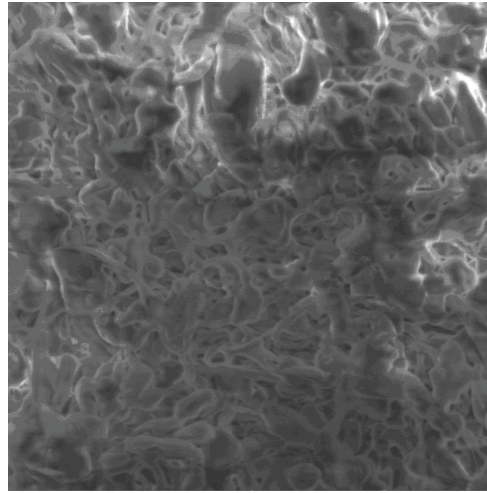


FIG. 3



c

FIG. 3: Structure of PTFE before (a) and after (b, c) mechanical activation ($\times 50,000$)

lar components, and each of them is intended for construction of certain morphological formations.

Thus, due to the high physicochemical and wear resistance indices, mechanically activated PTFE and its compositions may be used for manufacturing antifriction parts of machinery and equipment moving joints (Budnik et al., 2015c).

6. PREPARATION AND MODIFICATION TECHNOLOGY OF FIBROUS FILLER

The surface of CF is inert under usual conditions (Bijwe and Sharma, 2013). The PTFE composition with CF is a complex heterogeneous system with numerous surfaces of phase distribution.

The occurrence of physicochemical processes in boundary layers during composition formation makes significant contribution to the structure formation and, thus, to the major operating properties in such compositions (Lipatov, 1977).

The formation of reaction surfaces of PTFE matrix and CF is an important scientific and practical task and its solution guarantees obtaining and reproducing the planned property of the composite.

It was suggested to modify the surface of carbon fiber by different methods in order to provide technological combining of a carbon fiber with a polymer matrix in PTFE-based antifriction composite materials (Shelestova, 2015; Sirenko et al., 2008; Stepashkin et al., 2014). Such filler processing allows increasing composite materials characteristics, which depend greatly on the adhesive bonds of carbon fiber and PTFE matrix (Lipatov, 1977).

The carried out research has shown that the most widespread method of carbon fiber surface modification with the purpose of improvement of PTFE adhesion to CF is thermal-oxidation of fiber surfaces. The thermal-oxidative procession of CF surface results in the increase of the specific surface and degree of adsorption (Budnik and Burmistr, 2009).

The characteristic features of thermal (before milling) and thermomechanical (during milling) modification of the fiber surface were studied from the results of experiments. Table 5 contains the research results of wear resistance of CFRP containing a heat-treated fiber, which proves the effectiveness of thermal (increase of wear resistance up to 100%) and thermomechanical modification (increase of wear resistance up to 130%) of CF.

The most effective thermal and thermomechanical modification is at 400°C (the temperature of crystallites melting PTFE is 325–350°C), it happens as a result of the change in the supermolecular structure, because of the increase in the flexibility of the PTFE macromolecules. The increase of strength by 50% and wear resistance by more than 2 times is observed for CFRP containing CF after thermomechanical modification.

The electron microscopy data (Fig. 4b) signify the essential change in the CF surface character after thermal modification. To intensify the energy effect during milling of CF, the mechanical modification was carried out in vacuum ($p = 550 \pm 10$ mm Hg). It was found that the strength of CFRP containing fiber modified in vacuum increases almost by 50% and the wear resistance increases by 100% while milling of CF in vacuum (Table 6).

The modern material science widely uses a synergetic approach to the issue of increasing of composites strength, thus, the research of combined influence of thermomechanical processing and vacuum processing of CF on the physicomaterial and tribotechnical properties of CFRP (Table 7).

As it was shown in Table 7, the strength of CFRP increases by more than 75% and wear resistance increases by more than 3 times, compared to an unmodified sample during thermomechanical modification of fibers at 400°C for 15 min in vacuum ($p = 550 \pm 10$ mm Hg).

Thus, having analyzed a series of experiments, revealing the effectiveness of the influence of different technological methods on composite properties, it turned out

TABLE 5: Wear intensity of CFRP ($I \cdot 10^{-7}$ mm³/N·m) on introduction of CF after thermal and thermomechanical modification

Modification	Control	Temperature, °C					
		100	200	300	400	500	600
Thermal	12.5	12.0	10.0	8.0	6.0	12.5	13.0
Thermomechanical		11.0	9.0	7.0	5.5	11.5	–

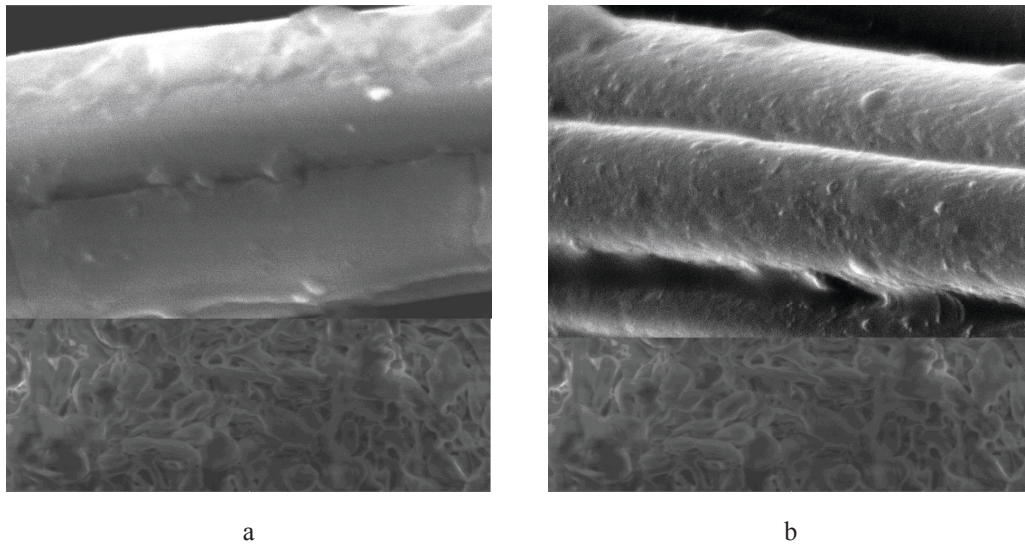


FIG. 4: Scanning electron microscopy microphotographs of the CF surface: a) before heat treatment ($\times 7500$); b) after heat treatment (400°C , 15 min) ($\times 9500$)

TABLE 6: Physical and mechanical properties of CFRP depending on the conditions of fiber grinding (grinding time 15 min, vacuum $p = 550 \pm 10$ mm Hg)

Parameter	Control	Rotational speed of operation, min^{-1}			
		7000		9000	
		Environment			
		Air	Vacuum	Air	Vacuum
Tensile strength, MPa	15.0	20.0	22.0	21.0	22.5
Wear intensity $I \cdot 10^{-7}$, $\text{mm}^3/\text{N}\cdot\text{m}$	12.5	7.0	6.0	7.1	6.5

TABLE 7: Properties of CFRP on thermomechanical modification of fiber grinding under vacuum conditions ($p = 550 \pm 10$ mm Hg)

Parameter	Control	Temperature of processing, $^{\circ}\text{C}$				
		100	200	300	400	500
Tensile strength, MPa	15.0	24.8	24.9	25.2	26.0	24.4
Wear intensity $I \cdot 10^{-7}$, $\text{mm}^3/\text{N}\cdot\text{m}$	12.5	4.4	4.4	4.2	4.0	4.5

that the most effective was thermal vacuum technology of CF modification, which allows increasing the strength of PTFE composite by 18–22% and its wear resistance by 20–25%, all these present practical interest for industrial implementation.

7. CHARACTERISTIC FEATURES OF MIXING TECHNOLOGY OF COMPOSITION INGREDIENTS

It is difficult to obtain a strong bond between the PTFE matrix and CF using traditional methods of filler preparation. The CF surface has an insufficient effect on polytetrafluoroethylene wetting; moreover its surface is hydrophilic that prevents physical and chemical sorption of macromolecules of the PTFE matrix.

The surface processing of carbon fibers promotes their interaction with the composite matrix (Shelestova, 2015; Budnik and Burmistr, 2009; Shelestova, 2006). When the traditional method of obtaining a carbon fiber filler (Sirenko and Budnik, 1992) with length distribution according to certain dependences is used, the carbon cloth is milled in a hammer mill and the cloth fibers separated by sieve are subjected to milling by the mill with bottom knives. The parameters of the milling technological process guarantee the distribution of fiber ensemble of certain length. But CF of fractional composition less than the "critical" length do not practically mix with PTFE, creating a dust containing agglomerate in composition volume and making longer, reactive fibers greasy and decreasing their compatibility with the PTFE matrix. It results in obtaining polymer composites with an insufficiently high strength and wear resistance indices, which is conditioned by the sufficient structure heterogeneity of the created composite.

The perfection of the method of carbon filler obtaining is possible due to the successive milling of CF in the presence of PTFE powder, allowing binding of fiber with the length less than "critical" in a CFRP agglomerate and preventing "agglutination" with longer fibers. Such qualitative and chemically reactive carbon fiber filler increases the strength and wear resistance of the PTFE composite (Budnik et al., 2009c).

The structure of CF, prepared *in situ*, is studied with the help of scanning electron microscopy. As the microphotography shows, there is a layer with increased content of PTFE on the surface of CF (Fig. 5).

The research analysis of experiments of composition with CF, prepared according to the above-mentioned scientific and technical solution (Budnik et al., 2009d) and known technological process (Sirenko and Budnik, 1992), shows that PTFE-based compositions obtained with the help of such a filler increase the breaking strength by 10–20% and the wear resistance by 17–40%.

The effect of increase of the composite operational properties due to the developed scientific-technological method consists of mechanical combining of small (powdery) CF with PTFE powder which results in creation of disperse composite product before the creation of composition in general. The composition will consist of three fractals at the end of volume structured skeleton formation. The first fractal is flour

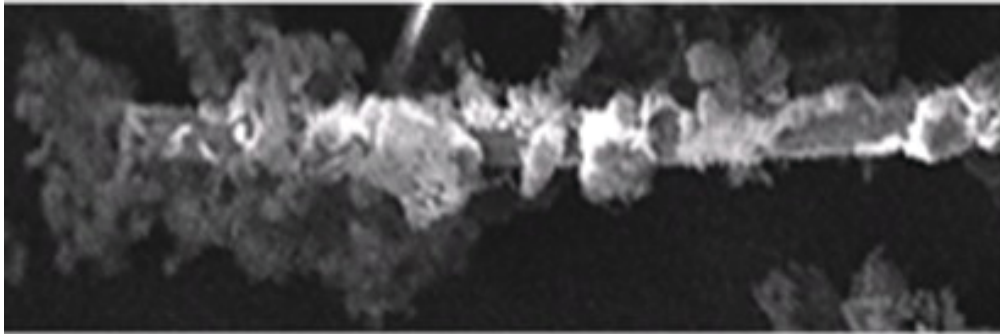


FIG. 5: REM microphotograph of the surface of CF modified by technology *in situ* ($\times 1300$)

particles of CF bonded with PTFE powder, the second is longer CF coated by PTFE (Fig. 5), the third is unbound mass of matrix PTFE. According to the percolation theory (Novikov et al., 2004), such a structure of polymer composite is a prerequisite of creation of an infinite cluster of filler (CF) in the polymer matrix (PTFE). Thus, the thermodynamic bond of small particles of fiber with necessary and sufficient volumes of PTFE powder results in obtaining of a more reactive filler, which has higher thermodynamic compatibility in comparison with a mechanical mixture of components in analogous proportions. It is confirmed by the data listed in Table 8. The introduction of CF into PTFE composition thus prepared sufficiently strengthens it and increases the wear resistance (Berladir et al., 2016).

To increase the effectiveness of mixing PTFE with milled CF, the influence of regime staging of mixing of CFRP components was studied (Table 9). The two-stage process is recommended. The milling of CF with mixture in proportion (mass) 1:1 is carried out *in situ* on the first stage; the necessary additional quantity of PTFE is introduced on the second stage (optimal proportion is 1:4) (Budnik et al., 2009a). [Q18]

As the data of Table 9 show, the strength properties of carbon fiber-reinforced plastic are increased by 45% and the wear resistance is increased by 80%, while producing the composition using two-stage regime in comparison with the verification regime.

TABLE 8: Physical, mechanical, and tribotechnical properties of CFRP obtained by traditional and authors' technology

Parameter	Traditional technology (Sirenko and Budnik, 1992)	Authors' technology (Budnik et al., 2009a)
Wear intensity $I \cdot 10^{-7}$, $\text{mm}^3/\text{N} \cdot \text{m}$	8.2	4.9
Breaking strength σ_b , MPa	18.0	24.0
Ratio of CF and powder of PTFE (volume) in the preparation of filler	–	1:1

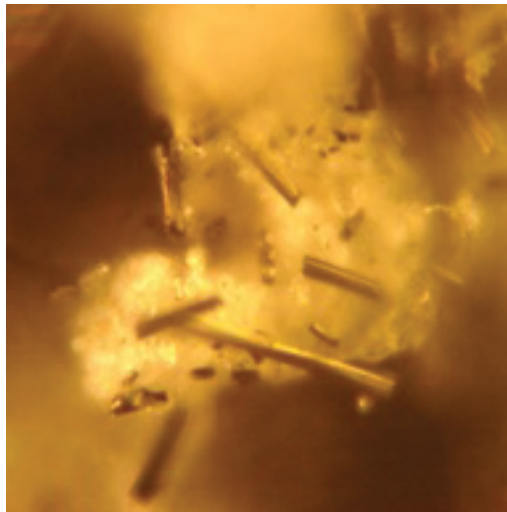
TABLE 9: Composition and properties of CFRP in the one- and two-stage mixing modes

Parameter	Control	Mode		
		One-stage mixing	Two-stage mixing	
			Stage I*	Stage II
<i>Composition (wt.%)</i>				
PTFE	100.0	80.0	20.0	60.0
CF	–	20.0	20.0	–
The mixture after I stage	–	–	–	40.0
<i>Properties of CFRP</i>				
Tensile strength, MPa	15.0	20.0	–	22.0
Strength at compression, MPa	28.0	31.0	–	35.0
Relative elongation, %	20.0	20.0	–	45.0
Wear intensity $I \cdot 10^{-7}$, mm ³ /N·m	12.5	9.0	–	7.0

* *in situ*

The positive effect is provided by "bonding" of CF flour particles (2–60 μm) by PTFE and creation of discrete power active centers of such a composition with high physicochemical activity as a result of mechanical activation, which is proved by the methods of electron microscopy (Fig. 6).

Due to the high strength and wear resistance indices, the polymer composition can be used for production of frictional unit parts of energy, chemical, and specialized equipment.

**FIG. 6:** REM microphotograph of CFRP obtained in two-stage mode, ×200

8. CHARACTERISTIC FEATURES OF THE TECHNOLOGY OF COMPOSITION MOLDING

One of the main drawbacks of PCM on the basis of PTFE and CF remains moisture absorption during operation of chemical and oil equipment with liquid and gaseous media (Sirenko, 1985).

The materials of the parts, operating under such conditions, show intermittent growth of wear, sometimes even critical with time. The positive results as to reducing the moisture absorption can be reached due to the optimization of composition molding technology.

The provision of stable bond of fillers and PTFE matrix in a composite is reached due to the optimal structuring of PCM during molding. This index reflects the provision of necessary thermodynamic, kinetic, and mechanical compatibility of system ingredients, gaining maximal physical and mechanical interaction on the polymer–filler interface and homogeneity in macrovolume of the composite, minimization of the structure defects and moisture absorption of composites while operating in the conditions of high humidity (Budnik et al., 2009b).

The operation characteristics and operation life are reduced significantly (3–4 times) and the wear rate is rapidly increased (4–8 times) with increase in the moisture exposure to compressor sealing composite material. It requires a shutdown and unscheduled repair, which finally results in economic expenses (replacement of components, equipment stoppage expenses, etc.).

As a control sample, the composite F4CF20 with such composition (wt.%) was used: PTFE — 80%, CF — 20%, which was obtained according to the known technology (Sirenko and Budnik 1992). The industrial process of molding is realized in the following molding conditions — 40–45 MPa, at the molding speed — $0.5 \cdot 10^{-2}$ m/s and time of molding at max pressure — 300 s (Technical Specifications, 1989).

The study of the mechanism and nature of moisture absorption by CFRP and evaluation of property loss by the composition was carried out.

The research results showed that the surface microdefects can appear in CFRP during product manufacturing, which can develop into destroying cracks (Fig. 7b).

According to the analysis of research data, it was found that moisture absorption of the composite with CF must be adjusted by molding technology, which defines the structure, properties, and durability of the composite material.

The main ways of composite moisture absorption and preservation of its operation properties with the help of the considered technology were grounded. These are the choosing of the optimal molding conditions; previous processing of CF with PTFE particles; introduction of processing aids into CFRP.

It was estimated that the main technological characteristics which influence the components compatibility, structure, and properties of composite during its molding are the pressure, molding speed, and time of molding under pressure. These factors were taken as the main for experiment planning and development of mathematical model of molding (Thomas, 2011).

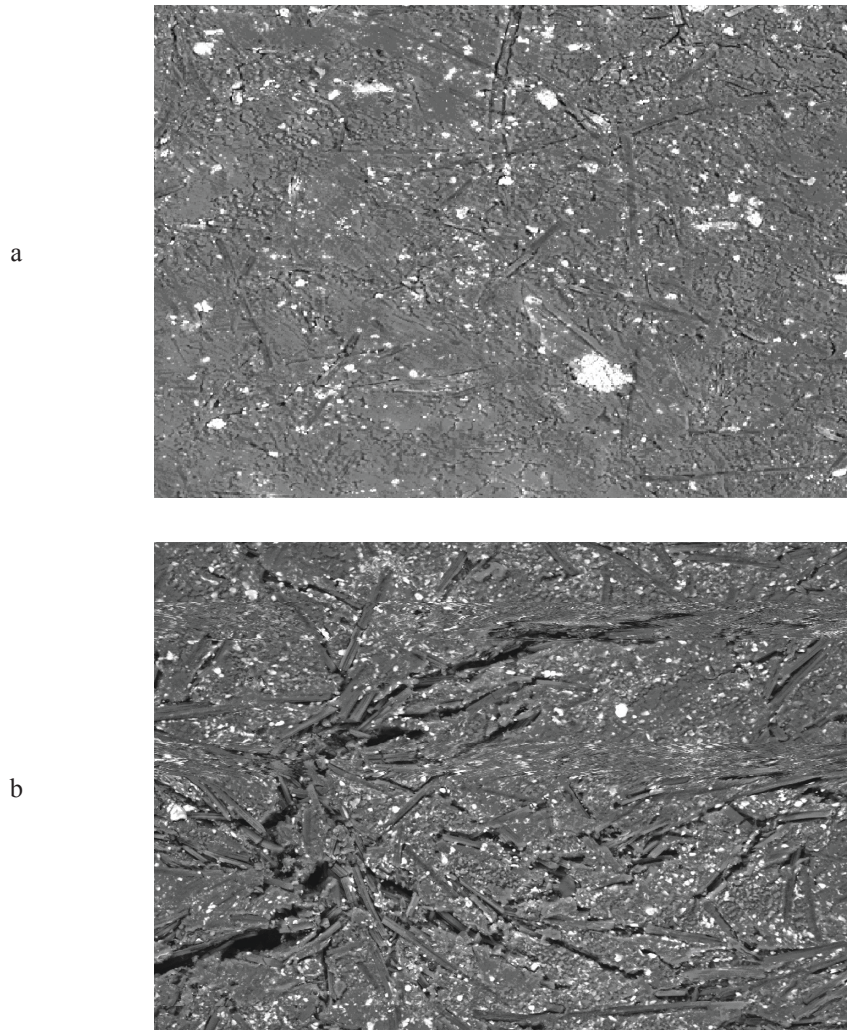
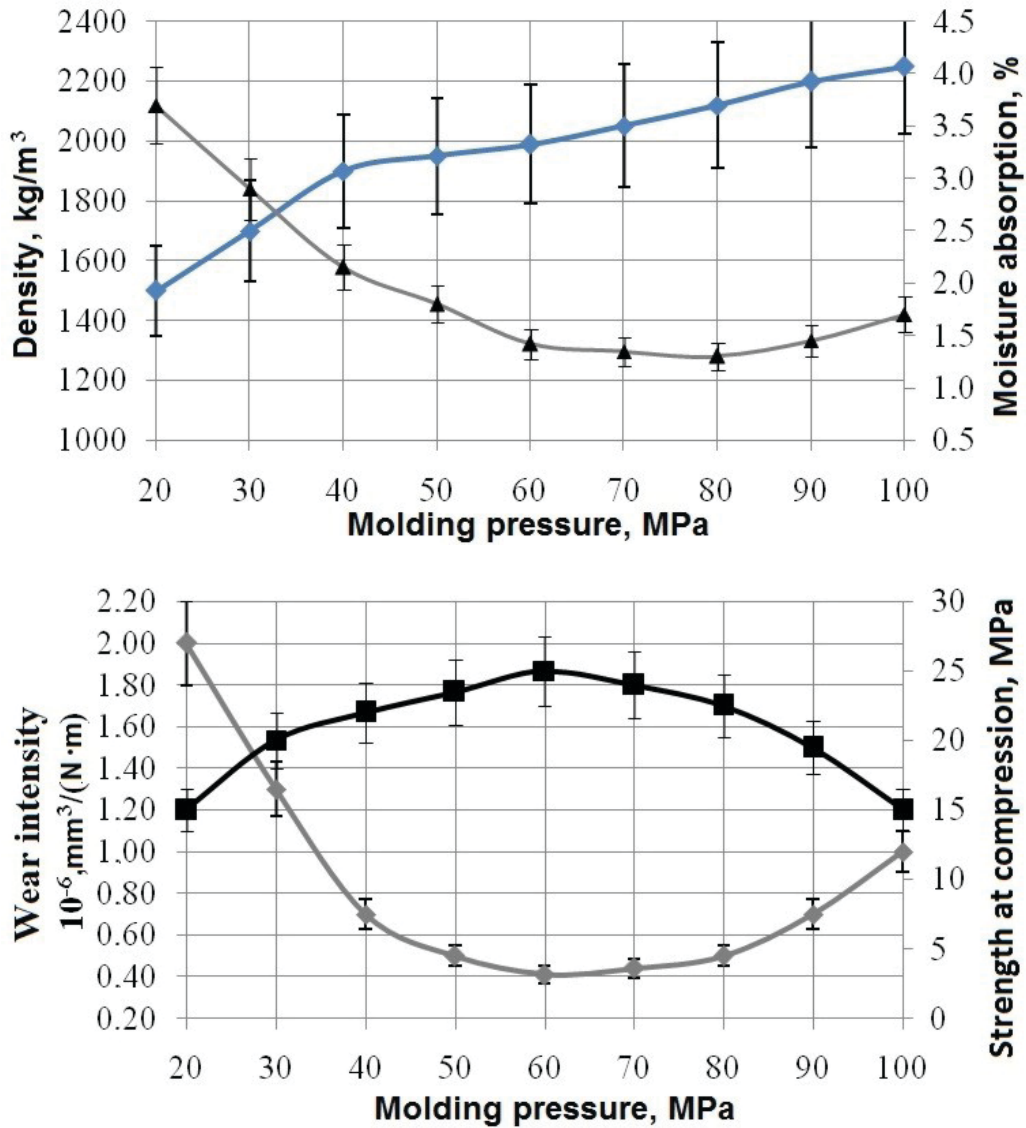


FIG. 7: Electron microphotographs of CFRP F4CF20 (Utracki and Wilkie, 2014) before (a) and after (b) exposition in water ($\times 230$)

To gain optimal functional characteristics of the PTFE composite, the crucial and main operation properties are the wear rate and strength boundary at a pressure that regulates operating capacity of frictional units in compression machines (Thomas, 2011).

The research results of the change of the physicomechanical and operation properties of composites depending on technological characteristics of molding are shown in Fig. 8.

It was found and proved by the research results, shown in Fig. 9 that the crucial factor that sufficiently influences the moisture absorption is the composite density.

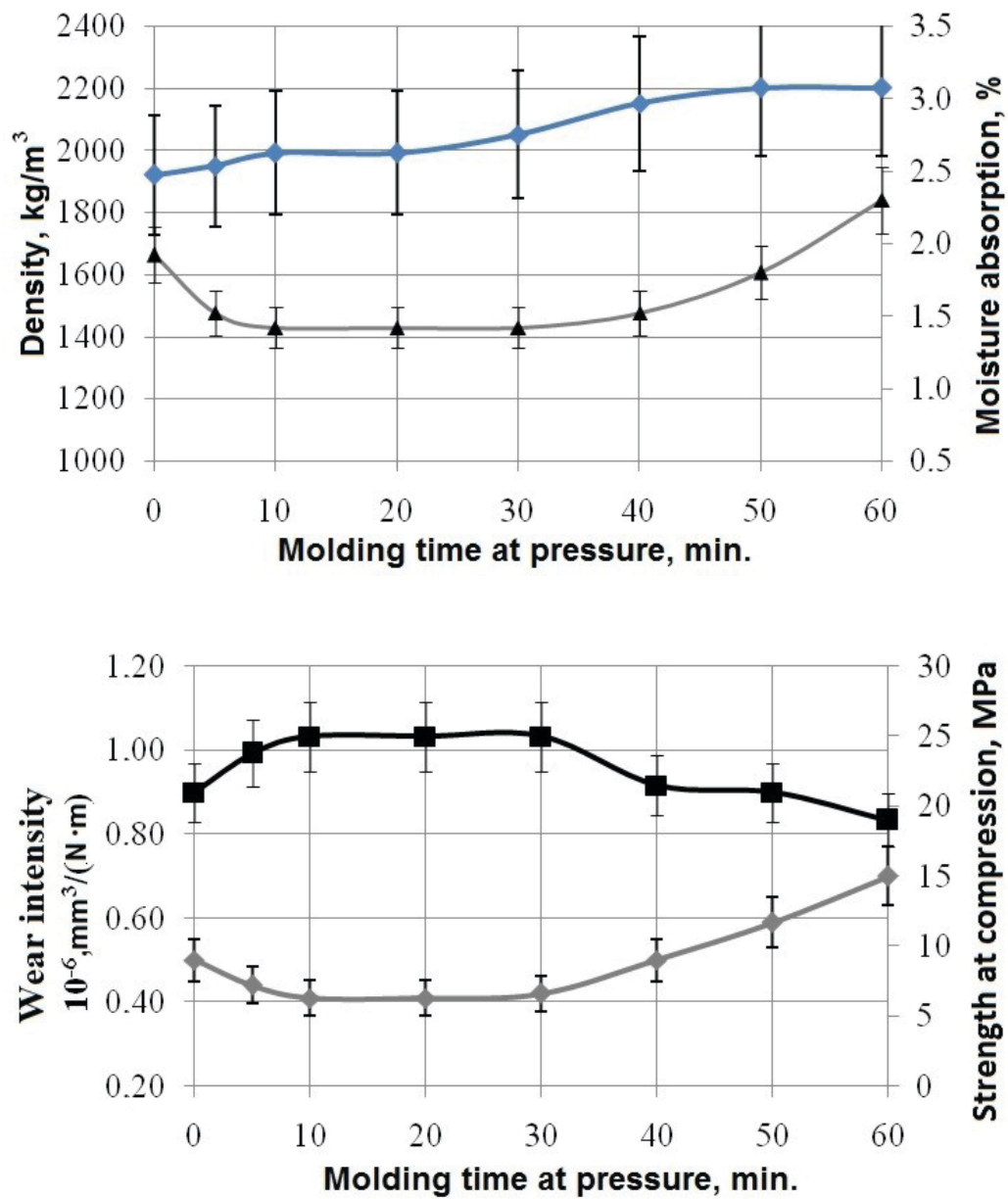


a

FIG. 8

The crucial technological factors are the molding pressure and molding speed in this case.

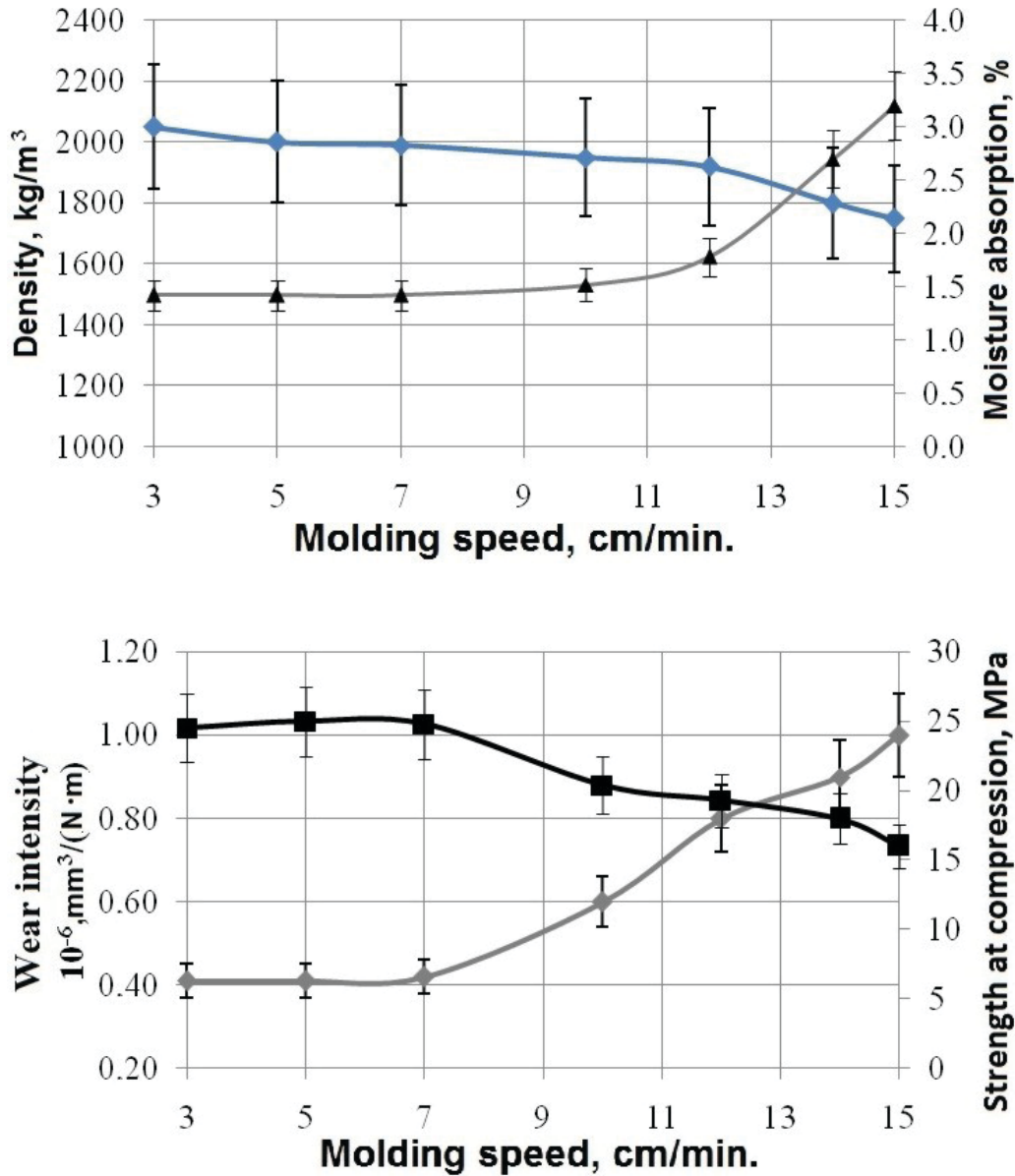
It was theoretically grounded and experimentally proved (Fig. 10) that optimization of the parameters of PCM molding technology contributes to the increase in the adhesive activity of PTFE matrix with CF.



b

FIG. 8

It is conditioned by the interaction activation of radicals on the interface and, as a result, moisture absorption is decreased and the physicomechanical and tribotechnical properties of PCM are increased.



c

FIG. 8: Dependences of the CFRP properties on molding pressure (a), time of molding under the maximum pressure (b), and molding speed (c)

Thus, the carried out research revealed and scientifically proved that the optimal conditions, providing stable phases of interaction of the PTFE matrix with

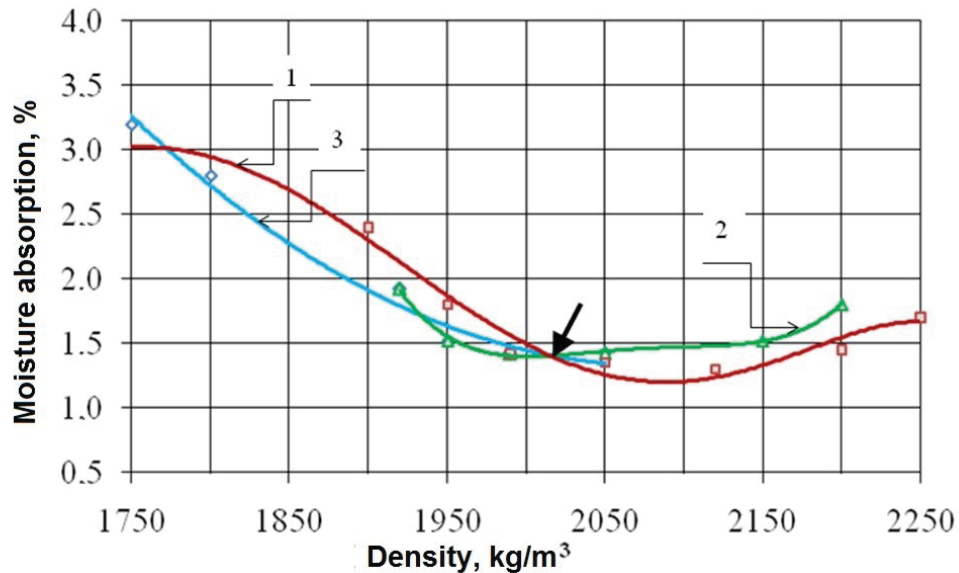


FIG. 9: Dependence of moisture absorption on density at a variation of parameters of pressing a composite: 1) molding pressure; 2) time of molding under pressure; 3) molding speed; \rightarrow optimum parameters

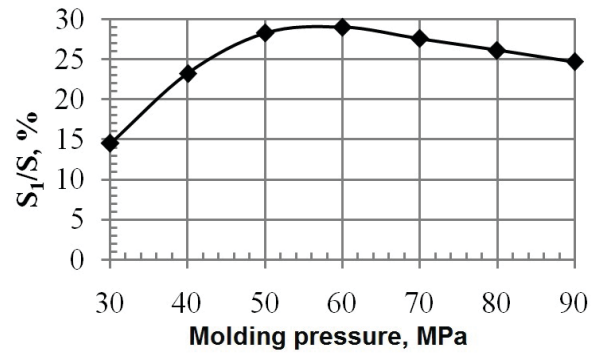
CF during molding as to physicochemical and tribotechnical properties are the following:

- molding pressure — 60 MPa;
- molding speed — 0.83 m/s;
- molding time at pressure — 600 s.

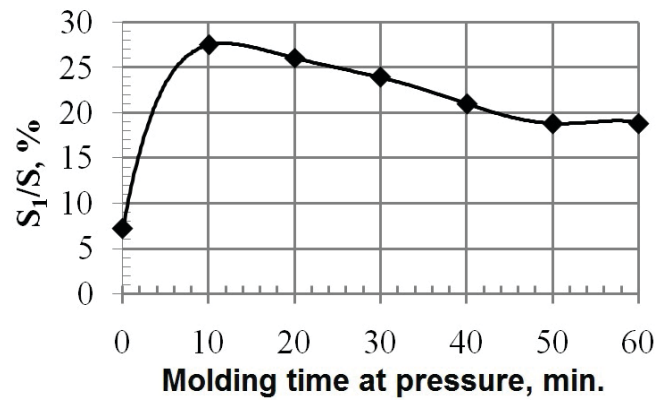
Such conditions provide CFRP with minimal moisture absorption (less than 15–20% in comparison with the analog) and high index of operation properties (compression strength is 15–25% higher, wear resistance is 40–45% higher than in the analog) due to the optimal structuring of CFRP during molding (Thomas et al., 2011).

9. CHARACTERISTIC FEATURES OF COMPOSITION SINTERING TECHNOLOGIES

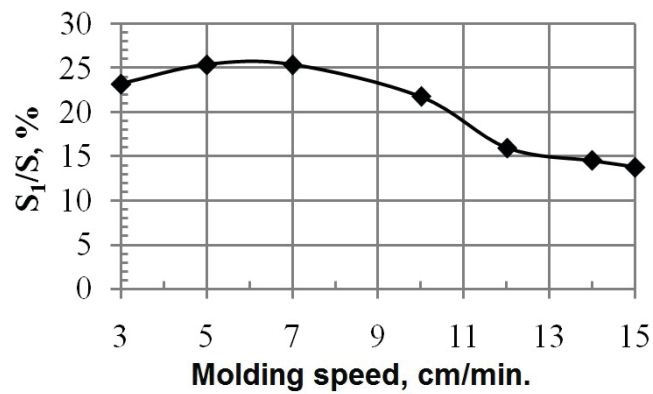
The service life of the composite parts depends on the heat exposure during their manufacture. The lack of sufficient information and summarized data about the impact of heat exposure on the properties of polymeric materials complicates the choice of optimal modes of production and heat treatment of PTFE composites (Drobny, 2009; Venkateswarlu et al., 2014).



a



b



c

FIG. 10: Adhesive durability of the CFRP depending on the technological modes of process of pressing (S_1 , surface area of ruptures; S , nominal area of the sample)

The technology of PTFE composites thermal processing is a thermal process of impact on the material that starts with the drying of the initial material and ends with the cooling of the heat-treated product (Pugachev and Roslyakov, 1987).

The main type of thermal effect on PTFE and its composition is sintering, which consists of heating of a half-finished product up to the temperature of 360–380°C, exposure at this temperature (1 h for 1 mm of thickness) and rapid cooling in the temperature range from 327°C to 350°C (Pugachev and Roslyakov, 1987).

The exposure time of the material during quenching has a significant effect on the properties of final products. This is due to the change in the polymer structure of macromolecules configurations and increasing of straight sections numbers which become PTFE cooling crystallization centers.

Thus, the technological modes of heat processing define the degree of crystallization and, as a result, they define the physicomechanical properties of the material. The crystallinity degree of sintered PTFE ranges from 50 to 70% and depends on the molecular weight and the speed of cooling. The majority of PTFE mechanical properties worsen with increase of the crystallinity degree (Venkateswarlu et al., 2014; Pugachev and Roslyakov, 1987).

The research basis is the task to increase the breaking strength and wear resistance of PTFE-based composite material, reinforced by CF, due to varying the sintering modes.

The assigned task is fulfilled due to the fact that composite material sintering is carried out in a cascade heat treatment mode, taking into consideration the time of phase transition and critical points of such transition of the composite material (Fig. 11) (Budnik et al., 2009c).

During sintering of billet material on the stage of particles fusion of polymer and carbon fiber filler, the chemical, physicomechanical, physicochemical, and electrical properties of the future product are formed in the composite. After applying the cascade heat treatment mode to the composite during sintering, which includes heating at different speeds and different time exposures with slow cooling, shift alternate disordered orientation, and structure stabilization on the molecular level take place. At the same time, the molecules fragments orientation takes place that allow forming a more homogeneous supermolecular structure and stable composite properties in the whole volume. As a result, the strength characteristics and wear resistance of the composite are increased.

The cascade mode of sintering with hour exposure on each step of cascade allows removing uncompensated tension of molding and provides complete structural transformations in composite volume during transition through characteristic temperature points of phase transformations, which prevents composite integrity damaging during cooling (cracking) and formation of necessary correlation of polymer matrix phases. All these allow forming the structure of CFRP of high homogeneity and stable properties of the composite. As a result, the strength and wear resistance characteristics of the composite are increased (by 25 and 50%, respectively) and it can be recommend-

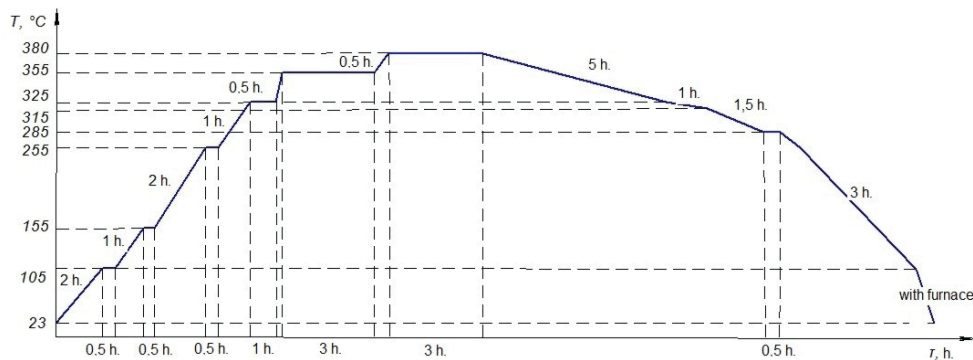


FIG. 11: Graph of cascade heat treatment during sintering of PTFE composite

ed for the production of construction materials of general and antifriction designation capable of operating at high temperatures and also in chemically active media.

10. CONCLUSIONS

The experience of many theoretical and practical works concerning the technology of PTFE composites formation, accumulated by many prominent specialists and the present authors in this sphere, has been analyzed and systematized.

The results of this work were published in many scientific editions, but the continuation of the search for the optimal technology of PTFE composites creation presents scientific and practical interest.

The research results defined operation regimes of process equipment: after the processing the PTFE composites have increased the operating properties in comparison with the basic ones and can be most effectively used for industrial implementation:

1. Energy impact on uncompounded PTFE results in the increase of the parameters of deformation and strength (breaking strength by 2.6 times, breaking elongation by 4.3 times) while preserving the high tribotechnical characteristics.
2. It turned that the most effective was thermal and vacuum technology of CF modification allowing the increase of PTFE composite strength by 18–22% and its wear resistance by 80 %.
3. While manufacturing the PTFE composite with CF using a two-stage mode, the level of strength characteristics of carbon-filled plastic in comparison with a control sample increases by 45% and the wear resistance increases by 80%.
4. The optimal molding modes were found that provide stable phases interaction of PTFE matrix with CF with minimal moisture absorption (less than 15–25% in comparison with the analog) and high operation properties (compression strength is higher by 15–25%, the wear resistance is higher by 40–45%).

5. The cascade mode of sintering of PTFE composite allowed obtaining PCM, which is characterized by the increased strength and wear resistance (25 and 50%, respectively) in comparison with the analog.

Thus, the PTFE composites are a bright illustration of material science triad, i.e., "composition–structure–properties". Although, the processes of the strengthening of such materials cannot be described with the help of the universal theory, but the technology of PTFE composites of tribotechnical designation has taken its place and is one of the main.

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