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Implementation of the Concept of Energy and Technological Compliance of Components in the Technology of Fluorocomposites

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Abstract: Structural and technological aspects of obtaining and processing functional composite materials based on PTFE (polytetrafluoroethylene) are considered. It is shown that, due to the features of the molecular structure of the matrix polymer, within the framework of the traditional technological paradigm, prerequisites are created for the implementation of a structural paradox, which manifests itself in a decrease in the parameters of the stress-strain and tribological characteristics of composites with an increase in the degree of filling. Within the framework of the concept of multilevel modification, methodological approaches to the implementation of the energy and technological compliance of components, which reduce the negative impact of the structural paradox, are considered.

Key words: PTFE, fluorocomposites, structural paradox, multilevel modification, energy and technological compliance of components.

1. Introduction

Composite materials based on PTFE (polytetrafluoroethylene) and other fluorine-containing matrices belong to the class of composites used for the manufacture of elements of metal-polymer systems, operated mainly under the influence of negative factors—high temperatures, without lubrication supply, aggressive media, etc., whose high demands on reliability and safety are placed to. This type of systems includes sealing and tribological elements of machines, mechanisms and technological equipment used in the enterprises of the chemical industry, heat power, processing industry [1-6].

The design of functional metal-polymer systems determines their service life under the action of not only tribological (tribochemical) processes, but also aging processes, thermal-oxidative degradation, deformation, fatigue failure, etc., that form the prerequisites for the manifestation of a negative synergistic effect. Therefore, the composite material for the manufacturing of a structural element for a metal-polymer system with high service life should include a set of modifiers with different mechanisms of action on the structure parameters at different levels of organization. This aspect is the rationale for implementing the principle of multilevel modification proposed by Kravchenko et al. [7] and developed in our studies [8, 9]. This methodological principle makes it possible, using the concept of a wear inhibitor proposed by Struk [10], to develop composites with an optimal structure that acts as a component of a metal-polymer structure that prevents (inhibits) the process of operational destruction for a certain period.

For implementing the principle of multilevel modification in PTFE-based composites, it is necessary to take into account the peculiarities of the

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structural and technological parameters of this type of thermoplastic binder, which distinguishes it from other types of industrial thermoplastics—polyamides, polyesters, polyacetals.

As follows from Ref. [4], there is an established opinion about the unique combination of stress-strain, physicochemical, thermophysical and tribological properties inherent in fluorine-containing materials, primarily PTFE, which favorably distinguishes them from other types of polymeric materials, mainly from thermoplastic ones. An analysis of studies [4] shows that these materials, as well as composites based on them, have a number of features that limit the areas of their practical application, primarily as wear inhibitors, in mechanical engineering. Among the most important features of the composition, structure and technology of fluorine materials and their composites, which negatively affect the expansion of the range of their practical application, in our opinion, it is necessary to include:

- the absence of a pronounced viscous-fluid state in PTFE at the melting temperatures of the crystalline phase, similar to the state of other thermoplastics, which makes it difficult to process composites into products using high-performance technologies,
- almost coincidence of the thermal degradation temperature and the melting temperature of PTFE, which determines the thermal-oxidative destruction of the surface layers of products and blanks during their sintering (monolithization),
- low activity of macromolecules of fluorine materials in the processes of adhesive interaction with solid-phase substrates of various compositions, which prevents the formation of a stable transfer layer that reduces wear,
- high temperatures and duration of the process of monolithization of compacted powdery semi-finished products based on PTFE, leading to high energy consumption and a decrease in the characteristics of the surface layers of products,
- sufficiently high friction coefficient leading to increased wear, during operation at high pV factor values, including the use of lubricants based on natural and synthetic components,
- low wear resistance of friction parts that reduces the range of application in friction units with high service life (especially it is typical in a low content of fillers and modifiers),
- macromolecule inertness to adsorption and chemisorption interaction with surface layers of modifiers with different consist, structure, habit and dimensions.

As noted in Ref. [4], these features, which are mainly due to the chemical structure of the molecular chain, the spatial configuration of macromolecules and their supramolecular packing, significantly limit the areas of practical application of products made from fluorine materials, primarily PTFE and composites based on it. Also, these features increase the energy intensity of the production and processing process and its cost. It is necessary to emphasize the pronounced inconsistency of the characteristic properties of fluorine-containing matrices, which are often mutually exclusive when creating composites based on them. For example, the easy movement of supramolecular aggregates and microvolumes of the PTFE matrix in comparison with other types of polymeric thermoplastic matrices during tangential shear causes a low friction coefficient of the metal-polymer pair during operation without lubrication.

Also, this property of PTFE, due to the peculiarities of the chemical structure of macromolecules, leads to high wear of the elements of the tribological system and the phenomenon of their cold flow under load. The low adhesive activity of PTFE layers and fluorine-containing oligomers, which imparts high hydrophobicity and resistance to active media, prevents their interaction with solid-phase fillers and the formation of a tough boundary layer, which contributes to the implementation of high stress-strain characteristics.

With all the varieties of brands of fluorocomposites (materials of the Flubon, Fluvis, F4K20, F4G10 series, etc.), when they are obtained, the general technological principle of formation and processing into products is implemented, which involves a combination of operations of mixing components in specified ratios, cold pressing of blanks and their hot sintering (monolithization) in an air atmosphere according to a given temperature-time regime. This technological principle, close in essence to that used in the technology of powder metallurgy, currently dominates in the literature, patents, and commercial sources, having become the basis of the technological paradigm of functional fluorocomposites [1-6].

The use of various technologies, consisting in the introduction of highly dispersed fillers (carbon-containing, silicon-containing particles: UDD, zeolites, etc.), including nanosized and mechanically activated [1-6, 11-13], as well as reinforcing fibers (CF (carbon fibers), glass, basalt, aramid) or their blends [13], while maintaining the traditional sequence of technological operations for obtaining blanks (products, technological paradigm) does not allow achieving fundamentally new effects of increasing the parameters of stress-strain, thermal and tribological characteristics.

The manifestation of a structural paradox for fluorocomposites, consisting in a significant decrease in the values of a number of important parameters (tensile strength σ_{uts}, friction coefficient f, density ρ) with the introduction of high-strength reinforcing fillers (for example, CF), is generally recognized [6, 13].

The purpose of this study was to develop methodological approaches to improving the structural parameters of the stress-strain and tribological characteristics of products made from them used in metal-polymer systems of various designs.

2. Materials and Methods of Research

Composite materials based on industrial PTFE of F-4PN, F-4PN90, F-4TM grades were used as an object for research.

Dispersed fragments of CF, obtained by grinding carbon tapes grade LO-1-12N produced by JSC Svetlogorsk Khimvolokno we used, were to modify the polymer matrix.

Highly dispersed particles of carbon black grade P234, UPTFE (ultrafine polytetrafluoroethylene), commercially produced under the Forum trademark, was used to implement the principle of multilevel modification.

Experimental samples were formed according to traditional and original technologies using the equipment of a specialized enterprise.

The structural characteristics of the fluorocomposites were studied by IR (infrared spectroscopy) spectroscopy (Tensor 27, Brucer), optical (Micro 200T-01), atomic force (Nanotop-3), and scanning electron (JSM-50 A) microscopy.

The parameters of the stress-strain and tribological characteristics of the model samples of fluorocomposites were studied according to common methods.

3. Results and Discussion

As noted by Avdeychik et al. [4], "the essence of the multilevel approach proposed by Kravchenko et al. [7] is the formation of the optimal structure of the composite material, which ensures the achievement of a given combination of product indicators from it. At the same time, the methods for creating such a structure are determined by the characteristic features of the molecular structure and mass and can be technologically implemented by introducing functional modifiers of various compositions, dispersion and activity into the matrix" [7].

For structural elements with high load-speed parameters, composites with a content of functional modifiers of 10-30 wt. % are used. These composites involve the development of special technologies for preparation, combination of components, formation of products and their modification, taking into account the specifics of the operation of a metal-polymer system of a particular design.

An algorithm for the formation of a multilevel optimized structure of composite materials with high performance parameters based on thermoplastic matrices with increased viscosity (PTFE) has been developed (Fig. 1). According to the algorithm, based on the methodological approach to controlling the processes of structure formation by directional changes in the parameters of the energy characteristics of the components, it is possible to create prerequisites for the formation of an optimized multilevel structure at the stages of preparation of components, their mix, formation of blanks and manufacture of products.

The features of the molecular structure of PTFE, described earlier, necessitate the search for new methodological approaches to minimize negative factors that prevent the formation of a low-defect structure with optimal organization parameters at various levels. Analysis of literature and patent sources devoted to materials science and technology of fluorocomposites containing functional modifiers in the concentration range of 1-20 wt. %, the results of which are presented in the monograph [13], indicates the presence of characteristic factors affecting the manifestation of the previously noted structural paradox, which manifests itself in the discrepancy between the parameters of the composite characteristics and the total parameters of the components with an increase in their concentration in the structure of the matrix polymer (Fig. 2).

When increasing the content of high-strength and wear-resistant modifiers, for example, CFs, a decrease in the total characteristics of the composite below the values corresponding to matrix PTFE is observed. An obvious reason for the manifestation of the phenomenon of the negative effect of modifiers on the structure parameters of highly filled fluorine composites (25-40 wt. %), the need to develop which is due to the requirements of modern mechanical engineering, is the energy mismatch of components at various stages of the technological process. In the nomenclature of the activation technology of components in order to form the necessary parameters of energy characteristics, mechanical activation technologies are distinguished. These technologies combine the effectiveness of action with sufficient simplicity and availability of technological equipment

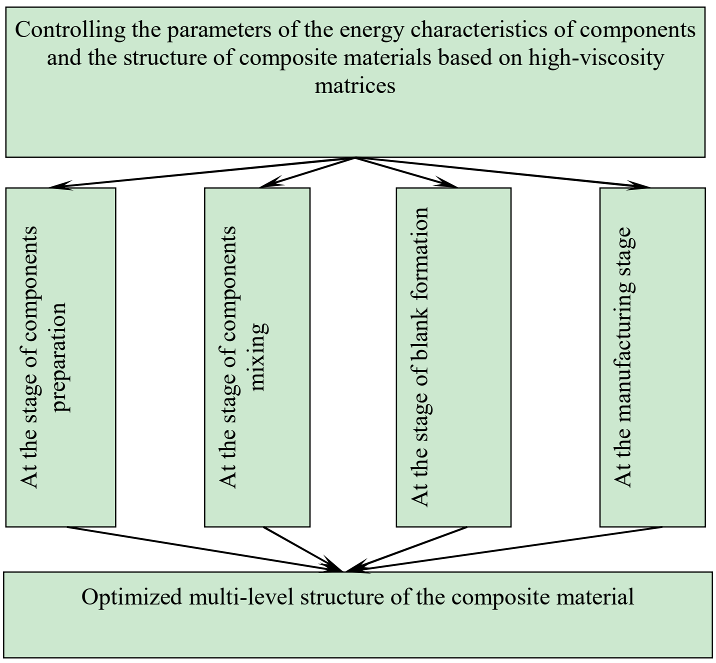


Fig. 1 Algorithm for formation of a multilevel optimized structure of composite materials based on thermoplastic matrices with high viscosity.

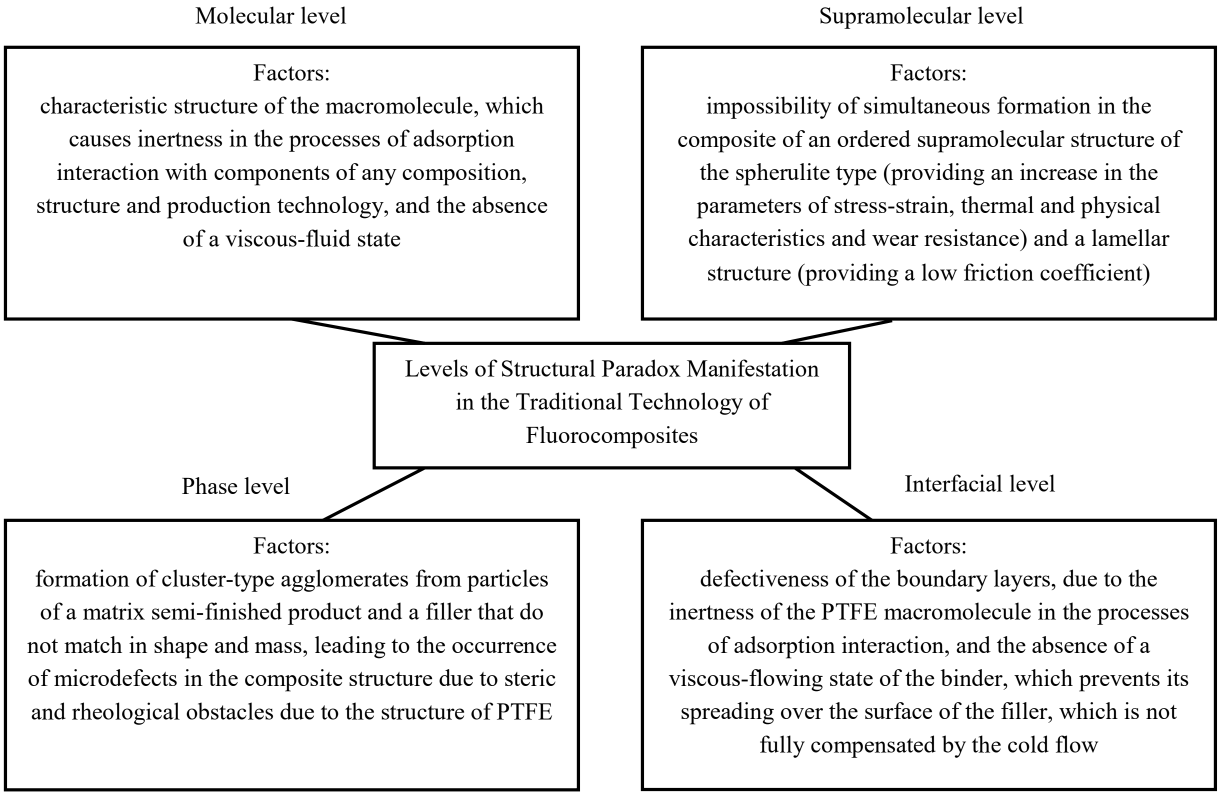


Fig. 2 Factors of manifestation of the structural paradox during the formation of fluorocomposites according to the technological paradigm [13].

for practical implementation. The mechanical activation of components makes it possible to achieve optimal energy parameters at a certain stage of the process and implement them during the formation of the structure at various levels of organization. A special role, as noted earlier, is played by the structure of the interfacial level, which plays a predominant role in the manifestation of a synergistic combination of strength, tribological, thermophysical characteristics of fluorocomposites, especially when the filler content is more than 20 wt. %. To implement the mechanochemical approach in ensuring the energy and technological compliance of the components, methodological principles for the technology of highly filled fluorine composites with increased performance parameters have been developed (Fig. 3). The most important component of the proposed principles is the joint activation of the surface layers of components using mechanical stresses on their combination both at the stages of mixing (MA (mechanochemical activation) technology) and at the stages of monolithization of the structure under high-temperature (TC (triaxial compression) technology) and low-temperature (CM (cold monolithization) technology) exposure [13].

As noted in our studies [8], the essence of the TC technology is the formation of a workpiece (product) from a composite material subjected to preliminary cold pressing in a closed form, which provides resistance to the thermal expansion of the components. The counteraction to the thermal expansion of the workpiece by the forming elements of the tooling is due to the difference in the thermal conductivity and thermal expansion of carbon steels and composition components. The technological equipment is designed in such a way that an initial tightness is created in the zone of contact between the workpiece and the mold wall. The design of the tooling may differ depending on the design of the product and the feasibility of individual contact surfaces hardening [13].

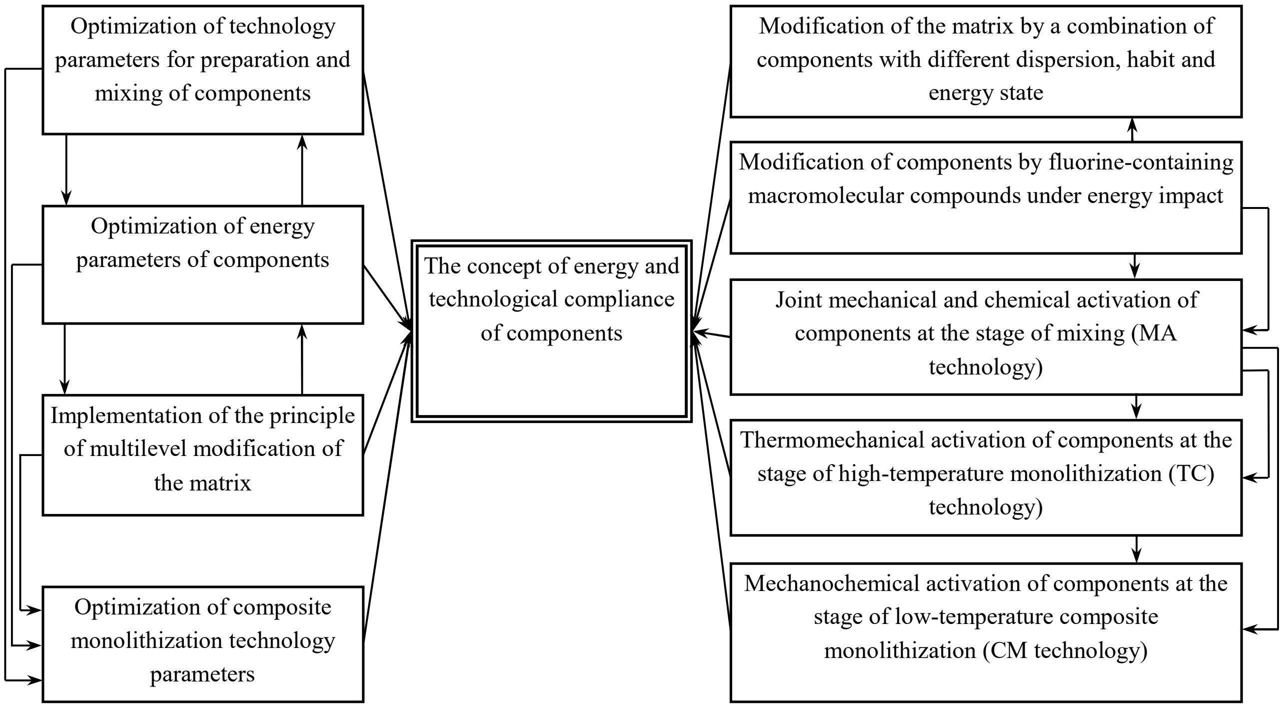


Fig. 3 Methodological principles of the technology of highly filled fluorocomposites with high performance parameters [14, 15].

The use of the developed TC technology for the formation of fluorocomposites makes it possible to reduce the defects due to the thermodynamic incompatibility of the main components—PTFE and CF.

Heat treatment of pressed blanks by free sintering does not form sufficient adhesive interaction at the “matrix-carbon fiber” interface, which leads to a low modifying effect of using a high-strength filler. The surface of the cleavage of the formed samples (Fig. 4) is characterized by numerous traces of tearing out of CF fragments from the matrix in the absence of adhesive interaction between the components (Fig. 4c).

Using the TC (triaxial compression) technology when forming composites, the number of structural defects decreases and the adhesive interaction at the PTFE-CF interface increases (Figs. 4b and 4d).

When using the developed method of mechanical activation of the components at the stage of their mixing (MA technology), homogeneity increases and the approximation of the shape (habitus) of the matrix particles (PTFE) and CF particles is ensured (Figs. 5c and 5d) compared to the traditional mixing technology (Figs. 5a and 5b).

A decrease in the degree of defectiveness and an additional orientation of CF fragments in the direction of mechanical stresses is observed in the process of implementing the developed technology of cold monolithization (CM technology) of blanks from fluorine composites (Fig. 6).

The specific pressure in the workpiece decreases due to the increase in the area of CFs, to which the compressive load is transferred, caused by the predominant orientation of their axes in a plane perpendicular to the direction of monolithization. In this case, the density of packing of CF fragments per unit volume of the billet increases, which leads to an increase in the resistance to axial load, and, consequently, to an increase in the compressive strength index of the sample at 10% deformation.

The developed method for manufacturing products (blanks) from fluorocomposites containing 10-40 wt. % CFs, which provides MA of the components at

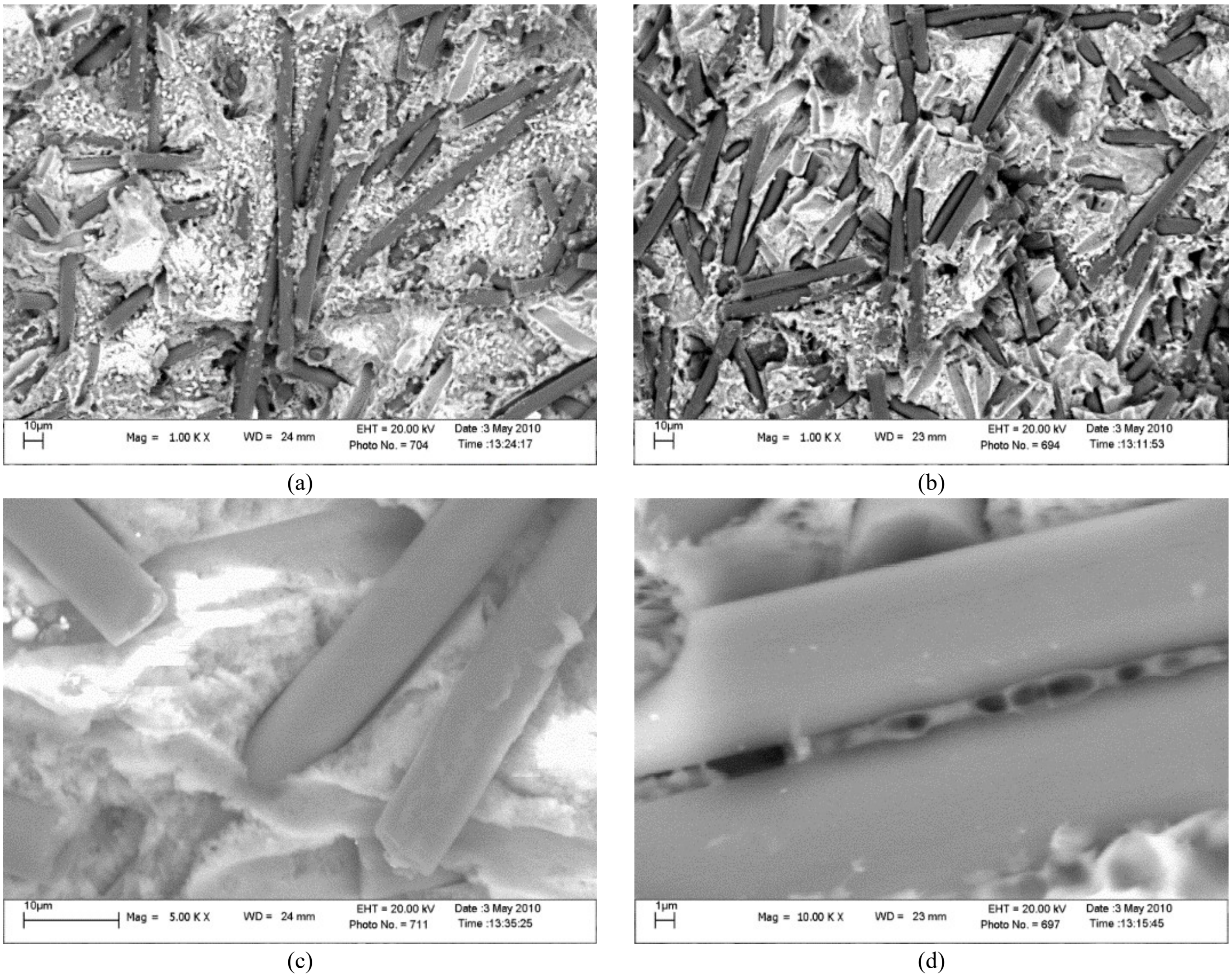
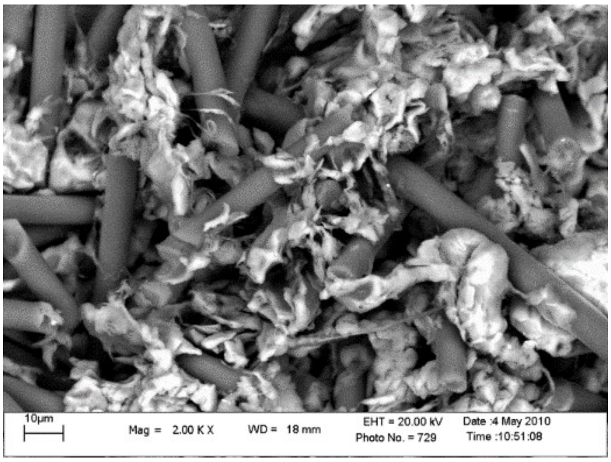


Fig. 4 Morphology of composite materials based on PTFE obtained by traditional technology ((a) and (c)) and TC technology ((b) and (d)).
Chips: in liquid nitrogen. The content of CF: 18 wt. %. SEM (scanning electron microscope): data.

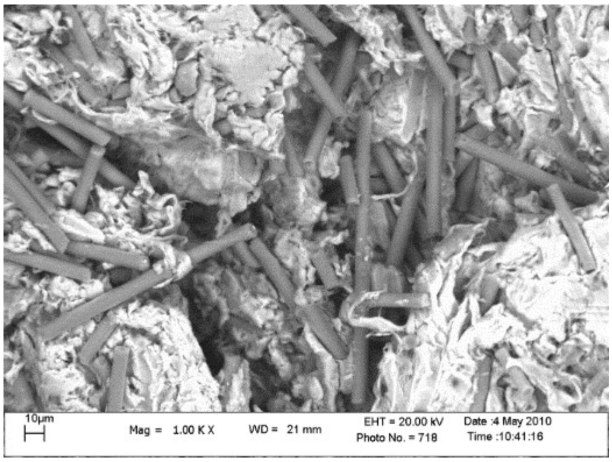
the final stage of the process, makes it possible to implement this approach in various versions: at the free sintering (monolithization) (version I); during sintering in a tooling, the inner surface of which is treated with a solution of a fluorine-containing oligomer (TC technology) (version II); during sintering in a tooling, on the inner surface of which a layer of polymer-oligomeric particles of UPTFE is deposited by the rotaprint method (version III); at the deforming a workpiece sintered in a tooling, on the outer surface of which a layer of a fluorine-containing oligomer is applied, in the tooling volume (version IV). In these versions of the developed method, the possibility of joint MA of the components at the final stage of the process is provided, which corresponds to the developed concept of energy and technological compliance of the components and increases the values of the parameters of composites characteristics (Table 1).

Developed on the basis of the concept of energy and technological compliance of components the technologies for obtaining fluorocomposites made it possible to reduce the negative effect of the structural paradox noted in Ref. [6] and manifested in a decrease in the parameters of characteristics at filling degrees exceeding 20 wt. % (Fig. 7).

A complex of studies of the features of the structure parameters of fluorocomposites with different content



(a)



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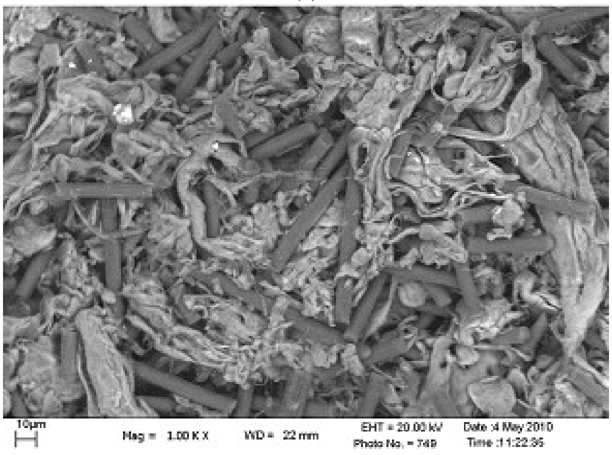
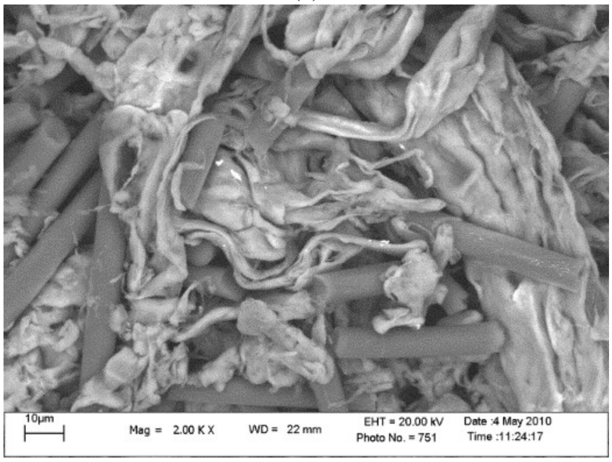
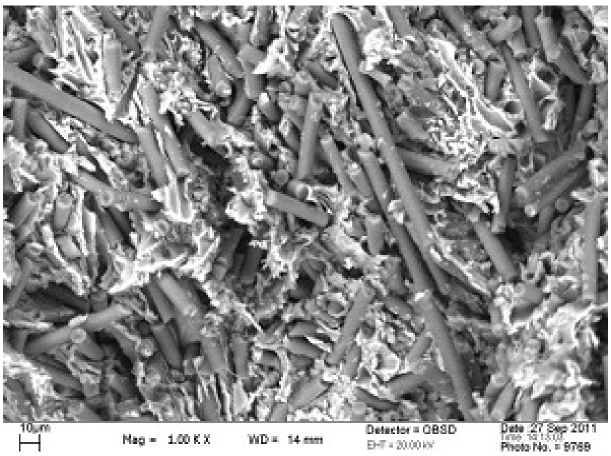


(c)

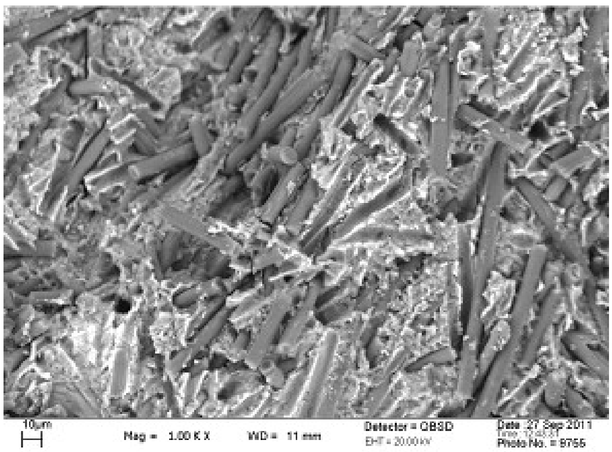


(d)

Fig. 5 Morphology of the mechanical blend of components of a composite material based on PTFE and CF, obtained using the standard technology ((a) and (b)) and the developed technology of MA technology ((c) and (d)).
SEM-data.



(a)



(b)

Fig. 6 Morphology of composite materials based on PTFE modified with CF during monolithization by sintering (a) and CM technology (b).
SEM-data.

Table 1 Parameters of the characteristics of highly filled fluorine composites during samples formation according to the traditional [16] and developed technology (CM technology) [17].

Characteristic	Parameter for the sample from composite				
	Formed by traditional technology [16]	Formed by the developed method [17]			
		Version I	Version II	Version III	Version IV
Density* (g/cm^3)	1.72	1.80	1.91	1.92	1.92
Strength at 10% compressive strain* (MPa)	14.0	28.0	30.0	31.0	46.0
Tensile strength σ_{uts}* (MPa)	15.0	17.0	21.0	22.0	27.0
Friction wear without lubrication** (10^{-7} mm^3/N·m)	4.7	2.0	1.6	1.5	1.4

* The content of the filler (CF "Viskum"): 30 wt. %.

** Tribological tests on a friction machine KhTI-72 at a normal load on 3 indenters 300 N, linear sliding speed on a steel disk 1.0-3.0 m/s.

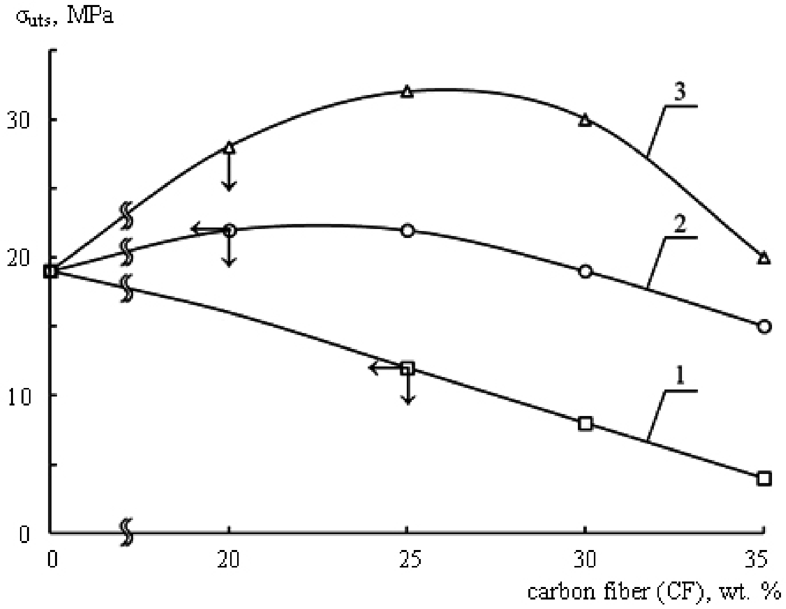


Fig. 7 Tensile strength σ_{uts} of PTFE composite materials in terms of the content of CF during mechanical combination of components (1), with MA of components and monolithization in a free state (CM technology) (2) and under conditions of TC technology (3) [6].

Table 2 Comparative parameters of the characteristics of composite materials based on PTFE.

Characteristic	Parameter for the composite							
	Flubon	Fluvis		Superfluvis		Obtained by developed technologies		
						Patent BY 9396	Patent BY 8480	Patent BY 9819
The content of the filler (wt. %)	30	30	20*	30	20*	20	20-40	20-45
Tensile strength σ_{uts} (MPa)	9	14	17*	18	27*	22-33	32-35	18-30
Friction wear without lubrication, *I*, (10^{-7} mm^3/(N·m))	5.0	5.0	3.5*	4.5	1.5*	1.5-2.3	1.3-2.0	1.7-1.9

* Data of normative technical documentation for materials "Fluvis" (TU RB 03535279.071-99), "Superfluvis" (TU BY 400084698.178-2006) of Grodno Mechanical Plant, JSC [18, 19].

of modifiers at different levels of organization, summarized in Ref. [13], made it possible to develop compositions of fluorocomposites with parameters of stress-strain and tribological characteristics that exceed the well-known analogues "Fluvis", "Flubon", F4K20 and others, and effective technologies for their production and processing into products for metal-polymer tribological and sealing systems, including those operated under extreme conditions (Table 2).

The developed technologies based on the activation of components during joint thermal, thermomechanical (TC technology), mechanochemical (MA and CM technologies) processing make it possible not only to achieve a synergistic combination of high performance parameters but also to expand the brand range of fluorocomposites, including highly filled ones, containing up to 40-45 wt. % of CFs and possessing high thermophysical and load parameters. The novelty of the developed methodological approach for implementing the principle of energy and technological compliance of components at various stages of the technological process of functional composites and the priority of domestic developments in the field of materials science and technology of fluorocomposites are confirmed by a series of patents of Russian Federation, Republic of Uzbekistan, Republic of Belarus [17, 20-24].

4. Conclusion

A systematic analysis of the influence of fluorocomposites structure on the mechanisms of deformation, destruction and wear of products made from them under various loading and operating conditions made it possible to identify the main factors influencing the manifestation of a structural paradox in the implementation of traditional technologies for their manufacture at the molecular, supramolecular, phase, and interfacial levels.

The studies carried out made it possible to propose effective approaches to the optimal choice of modifiers for high-molecular matrices, including PTFE:

(1) it is advisable to use highly dispersed particles, predominantly of the micron range size, with an extended morphology of the surface layer formed by nanosized components of various compositions and structures;

(2) the correlation of the geometric parameters of the components of the surface layer and the volume of a highly dispersed particle should be determined using physical criteria characterizing the nanostate of the selected material objects (matrix and modifier);

(3) to ensure an effective modifying action, a highly dispersed particle must be given a special energy state due to the combined action of structural-chemical, dimensional and technological factors. The choice of the prevailing factor for achieving this state is determined by a combination of operational, energy, economic, environmental parameters determining the effective and expedient industrial application in accordance with the terms of reference,

(4) when choosing the method of activation of highly dispersed particles that provide optimal modification, it is necessary to establish the prevailing mechanism for the formation of a transitional (boundary) layer of a given structure and parameters of stress-strain and adhesion characteristics and unconditional implementation of the principle of “reasonable sufficiency” in relation to a specific combination of materials science, environmental and economic factors;

(5) the greatest prospects for the creation of functional composite materials and their large-scale production based on high-molecular matrices are dispersed particles obtained from natural, synthetic and artificial semi-finished products using traditional and special technologies that ensure the formation of a surface layer morphology with nanosized components with an optimal level of energy activity;

(6) to obtain composites based on high-viscosity and high-melting matrices, it is preferable to use technologies for joint MA of components and multilevel modification using particles of different composition, structure, and dispersion.

(7) the formation of optimal structure of a nanocomposite material at a certain level of organization is possible on the basis of the concept of energy and technological compliance, which provides

a correlation of the energy parameters of the components sufficient for the prevailing physicochemical process to occur, which determines the parameters of the characteristics of the boundary layers, under certain conditions of technological impact on them at stages of preparation, mixing and processing.

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