STRUCTURAL PRINCIPLES OF FORMATION HIGHLY DURABLE TRIBOTECHNICAL MATERIALS BASED ON POLYTETRAFLUOROETHYLENE

СТРУКТУРНЫЕ ПРИНЦИПЫ ФОРМИРОВАНИЯ ВЫСОКОПРОЧНЫХ ТРИБОТЕХНИЧЕСКИХ МАТЕРИАЛОВ НА ОСНОВЕ ПОЛИТЕТРАФТОРЭТИЛЕНА

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Abstract: *The preconditions of forming a structural paradox within the existing technological paradigm, which manifests itself in reducing the parameters of strength and tribological characteristics of composite materials based on polytetrafluoroethylene when administered in their composition of fillers and modifiers of different composition and geometry when the content of 15-20 wt. % Established effects of forming the structural conditions derating improved performance due to the formation of cluster structure of the binder particles (PTFE) and the modifier. The effective technological methods to ensure reducing the likelihood of cluster components in the manufacturing process of highly composites with a filler content of 20-35 wt. %. The technology of producing high-strength wear-resistant fluorine composites, 1.5-20 times superior to common parameters analogues produced under the trademarks "Flubon", "Fluvis", "Superfluvis.". We consider the effective use of highly fluorine composites in mechanical engineering, chemical industry and energy.*

***KEYWORDS**: FLUORINE COMPOSITES, STRUCTURAL PARADOX, TECHNOLOGICAL PARADIGM, CLUSTER STRUCTURE, TECHNOLOGY FOR STRENGTHENING*

1. Introduction

Despite the unique combination of parameters of service characteristics - chemical resistance, heat resistance, low coefficient of friction, products of unmodified polytetrafluoroethylene (PTFE) limited use in the construction of friction units of machines and technological equipment [1-7]. For products of polytetrafluoroethylene characterized by increased wear during operation without an external supply of lubrication and high loading-speed modes, as well as a manifestation of cold flow, which leads to changes in the design parameters of the friction unit.

An effective way to increase the parameters of strength and tribological PTFE is the introduction of the matrix polymer fillers and modifiers of different composition, structure and mechanism of action and dispersion [1-6], as well as workpieces and articles by high flows, including ionizing radiation [6]. The last line, despite the possibility of regulating the parameters of strength and tribological characteristics in a sufficiently wide range of limited use due to technological difficulties of implementation.

In the nomenclature of composite materials of different functionality occupy a special place fluorocomposites, based on polytetrafluoroethylene modified disperse various components of the composition, structure and technology of [1-10]. Despite the existing range of branded fluorocomposites, providing the possibility of manufacturing structural, sealing, tribological and protective elements machines and process equipment with the given parameters of service characteristics, the problem of achieving the maximum values of the parameters of strength, wear resistance is far from the solution and requires the development of new technological approaches that take into account the specific structural features of the macromolecules of polytetrafluoroethylene (PTFE), conditional on the physico-chemical mechanisms and tribochemical processes in static and dynamic contact components and metal-filled materials systems.

Analysis of the literature devoted to the study of physico-chemical and technological aspects of the production and processing of functional fluorocomposites and the use of these products in various fields of technology, evidence of mature traditional methodological approaches based on classical concepts of polymer materials science, physics, chemistry and technology of plastics and composite materials [1-3]. The essence of these approaches is to apply the methods of regulation of the supramolecular structure of PTFE matrix by introducing a dispersed phase modifiers and composite structures - through the use of fiber fragments of organic and inorganic fibers - glass, oksalonovyh, basalt, carbon [1-3].

Filling materials based on PTFE - fluorocomposites - developed effective methods of managing structural parameters at different levels [2, 6, 8], which allowed us to develop and master the industrial brand variety, including a few dozen items with different parameters of strength, tribological and thermal characteristics [3, 7-10]. With all the variety of stamps fluorocomposites (Papers Series "Flubon", "Fluvis" F4K20, F4G10 et al.), In their preparation implemented common technological principle of forming and processing into products, involving a combination of the operations of mixing the components in a predetermined ratio, cold extrusion billets and their hot sintering (monolitizatsii) in air at a given temperature-time mode. This technological principle is similar in essence to those used in powder metallurgy, currently dominates the literature, patent and commercial sources, becoming the basis for technological paradigm functional fluorocomposites [1-3, 8-11].

The current technological paradigm functional fluorocomposites led to the achievement of a certain level parameters of strength and tribological characteristics which excess within it is not possible or is costly material and energy resources, reduces the effectiveness of the practical application of the products obtained [1-3, 8-12] . The inefficiency of traditional approaches in the implementation of technology of functional fluorocomposites particularly pronounced when creating filled materials containing more than 20% by weight. components of different composition and dispersion, which dramatically narrows the range of their practical application in tribological and sealing systems operating at high loading-speed and temperature conditions. The result is a scarce resource exploitation friction units of special equipment, vacuum, cryogenic plants and sealing elements of compressors for compressed and liquefied gases [3, 6-8]. Meanwhile, the analysis of the mechanisms of destruction and wear products from highly filled material indicates the potential of an incomplete as matrix polymer (PTFE) and modifying component (typically of high strength and wear).

The purpose of this study was to develop the principles of improving the technology of machine fluorocomposites based on the concept of multi-level modification proposed in [13].

2. Preconditions and means for resolving the problem

As a basic binder used in the preparation of fluorocomposites commercially available polytetrafluoroethylene (PTFE) marks F-4PN, F-4PN90, F-4TM differing average size of the powder fraction (JSC "HaloPolymer", Russia). For reinforcement PTFE matrix fragments used carbon fiber (HC) received mechanical dispersion of the carbon tape brand LO-1-12N (JSC "SvetlogorskHimvolokno", Belarus) with the size fractions less than 200 microns. Structural modification of PTFE was carried out in the introduction of carbon black (TS) grades P234 and P803 with a mean size of individual particles 20 and 80 nm, respectively, as well as polytetrafluoroethylene Ultrafine (UPTFE), an oligomeric polymer products termogazodinamicheskih synthesis polytetrafluoroethylene, obtained according to [6, 7]. Used in the experiments UPTFE, commercially available under the trade name "Forum" (Institute of Chemistry, Far East Branch, Russian).

Samples for studies of strength and tribological characteristics developed fluorocompositesprepared in accordance with the requirements of normative documents on materials such as "Flubon" (analogous to "Fluvis") [9, 10]. Defines the parameters of strength(σ_t , σ_{comp} , E_{comp} , HB) and tribological properties (wear intensity I, the coefficient of friction f) according to standard procedures or methods recommended in [9, 10], with the use of plants MR-200 ComTen 94c, P-0.5, HP-250, friction machines SMC-2, HTI-72. Analysis of physico-chemical and structural aspects of the modification of PTFE, technology acquisition and processing fluorocomposites and operating features of these products was carried out on the basis of IR spectroscopy (Tensor-27), nuclear power (NANOTOP-III), optical (Micro200T-01) , scanning electron (LEO1455VP) microscopy and X-ray diffraction (DRON-2.0).

Samples were prepared for research on technological regimes recommended in [3, 11] and by original technologies, realizing the possibility of multi-level modification.

3. *Results and discussion*

Analysis of the literature devoted to materials science and technology of functional fluorocomposites [1-13], suggests that the main provisions of the traditional technology, component paradigm is the use of classical methods of regulating the supramolecular structure of the matrix polymer using fillers dispersed organic and inorganic nature and the introduction of PTFE fragments of reinforcing fibers dispersed using mechanical mixing operations of components of cold pressing and monolitizatsiipreforms at temperatures above the crystalline melting point of the binder phase.

Using this technology, the various species, consisting in the introduction of fine fillers (carbonaceous, siliceous - UDD UDAG, zeolites, etc.), including nanoscalemechanoactivated [1, 4, 5], and the reinforcing fibers (carbon, glass , basalt, aramid), or mixtures thereof [3, 12], while maintaining the traditional process flow for obtaining blanks (products), not achieve a fundamentally new effects enhance the parameters of strength, thermal and tribological characteristics. Use of complex modifier comprising finely divided carbonaceous material fraction (cryptocrystalline graphite) in combination with dispersed reinforcing fibers of carbon fragments (HC) [2], the conventional technique of forming blanks not allowed to significantly increase the values of tensile strength, toughness and wear resistance, which is determined the potential for their use in the construction of machines and technological equipment with high demands on reliability and guaranteed life.

In some literature concluded impossible to maintain some of the initial parameters of the matrix polymer, polytetrafluoroethylene (σ_t example, specific toughness (NDC)) and increase their values upon administration of any composition and filler dispersibility, including high strength, especially at elevated levels of [8]. It is generally accepted structural paradox for fluorocomposites consisting in a significant decrease in the values of a number of important parameters (σ_t , f ,density) with the introduction of high-strength reinforcing fillers (eg, HC). An analytical expression is capable of evaluating the expression of the structural paradox when creating fluorocomposites [11]:

$$\sigma_{tc} = \sigma_{tm} - 0,5i \qquad (1)$$

where σ_{tc} - the composite tensile strength, MPa; σ_{tm} - Tensile strength of matrix polytetrafluoroethyleneMPa; *i*- the filler content,% wt.

From this expression it follows that the introduction of the composite of more than 20% by weight. anyone, including high strength, the filler is impractical because there is a significant decrease in the values of σ_t parameter that determines the field of application of its products.

Confirmation of the validity of applying the proposed in [11] analytical expressions (1) to estimate the parameters of service characteristics of engineering fluorocomposites are parameter values for σ_t most effective brands that have found widespread application - Flubon-20LO, Fluvis-20 F4K20 significantly inferior to similar values of the source of polytetrafluoroethylene (30-36 MPa) [3, 6-11] and are close to the calculated values, defined by the formula (1) (Fig. 1).

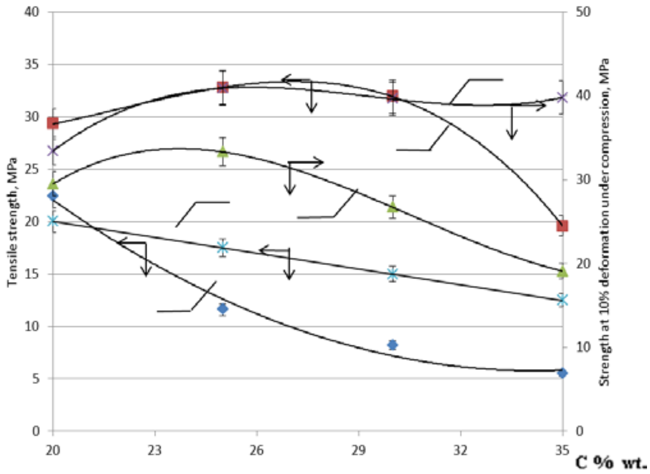

Fig. 1 *Dependence σ_t parameters (1, 2, 4) and σ_{comp} at 10% deformation (3, 5) the content of carbon fiber in the composition (C % wt.). Depending 1, 3, 4, 5 - experimental dependence of 2 - calculated according to [11]*

Several studies have analyzed the influence of the structure of the boundary layer at the interface level "matrix-filler" on the parameters of strength and tribological characteristics fluorocomposites sold under the brand name "Fluvis" (analogous to "Flubon") [2, 8]. Suggested the determining role of the bond strength at the interface in the implementation of the parameters specific σ_t and toughness, and the method of modification of carbon fibers by plasma treatment in an environment of fluorinated gases to increase the adhesion at the interface [8]. A similar approach was previously proposed in [3] the fibers during processing of low molecular weight fluorine compounds and methods of using the HC oxidation and etching. Achieved the effect of increasing the parameter σ_t using both the modified carbon [8, 9] and Arselon [12] confirms the validity of the fibers is not the hypothesis of the decisive influence of fluorine compounds on the surface of the reinforcing filler on the parameters of deformation and strength characteristics of composites based on PTFE. Thus, the parameter σ_t for materials such as "Flubon-LO", "Fluvis", "Superfluvis" does not exceed the values specified regulatory documents [8, 9], are, respectively, 9, 17 and 27 MPa, which is significantly inferior to the parameter value for the base σ_t polytetrafluoroethylene equal to 32 MPa [1, 6] (Table 1).

In addition, as the performances of arrangements of the process of polymerization of fluorine-containing monomers [2, 3, 6, 11], to form high molecular weight compounds from the gaseous medium must be compliance with the specific conditions that can not be realized to the extent required in the amount of vacuum in the process chamber modifying semifinished carbon fiber tape in the form [8]. Information set forth in [8] et al., did not confirm the formation of polymer products of similar molecular weight, composition and structure of polytetrafluoroethylene to a macromolecule.

Table 1 *- Parameters of service characteristics of highly fluorocomposites*

Parameter	Value for the material							
	Flubon-LO	Fluvis		Superfluvis		Technology MA	Technology HC	Technology CM
σ_t, MPa	9	14	17*	18	27*	26	30	32
$\sigma_{comp\ 10\%}$, MPa	27	30	-	33	-	35	40	45
$I \cdot 10^7$ mm^3/H • m	5,0	5,0	3,5*	4,5	1,5*	2,4	2,3	1,5
ρ, kg/m^3	1830	1830	1930*	1850	1990*	1870	1920	1940
The content of CF,% by weight.	30	30	20*	30	20*	30	30	30

* - These regulatory documentation for materials "Fluvis", "Superfluvis" [9, 10].

Probable mechanism of manifestation of a technical effect of increasing values of the parameter σ_t, when introduced into the PTFE carbon fiber modified by plasma method [8], is to change the strength of interparticle friction between the components of the dispersed material (PTFE and HC), helping to reduce clustering processes that determine the prerequisites for the formation of structural defects [6].

Thus, the experimentally observed adverse effect of the values of a number of important parameters fluorocomposites with the introduction of the high-strength fillers can not be explained using classical ideas about the role of supramolecular structures at the interface and in the implementation of mechanisms of deformation and failure of these products under the influence of operational factors.

However, for any type fluorocomposites containing superfine as including nanoscale [1, 2, 5] and the fibrous reinforcing fillers (original and modified) [2, 3, 6, 8], and mixtures thereof, [2] realized the effect of multiple parameter is increased wear resistance in friction products for metal counterbody without external supply of lubricant. Obviously, this effect is a consequence of the mechanism of manifestation of the filler particles create any composition, structure and mechanical dispersion of obstacles to deformation and displacement of local areas of the binder matrix under the influence of tangential stresses and their own resistance to tribological factors - thermal and mechanical.

Systematic analysis of the impact on the structure fluorocomposites deformation mechanisms, fracture and wear products from them in various loading conditions and operation revealed the main factors influencing the expression of the structural paradox in the implementation of the traditional technology of their production at the molecular, supramolecular, phase and interphase levels (Fig. 2).

Imperfection fluorocomposites structure at the molecular level due to the specific structure of the macromolecule polytetrafluoroethylene causes no plastic condition,characteristic of thermoplastics, which determines the ability of the matrix to the spreading over the surface of the filler and inert macromolecules expressed in the processes of physical and chemical interactions with components of any composition, structure, production technology.

The molecular structure of PTFE necessitates use of special procedures for the formation of products (semi-finished), providing interaction of individual particles of the dispersed matrix macromolecules by interdiffusion with the formation of boundary layers little defect structure at temperature influence on the compressed sample (mono politicization). This factor has caused the need for long exposure workpiece at temperatures exceeding crystalline melting temperature (330-350 ° C) to form a monolithic matrix with a minimum amount of defects, supermolecular structure which differs significantly from the structure of the starting powder particles semifinished. The surface layers of dispersed particles contained in mechanical contact of an external pressure at mono politicization play the role of active nucleating agent, resulting in the recrystallization process and the formation of supramolecular organization of a new type.

Therefore, the role of morphology and size of the dispersed particles of commercially available PTFE is very important for the formation of the structure of the matrix with the optimal combination of parameters of strength and tribological characteristics. Given that commercial grades of PTFE (F4, F4M et al.) Very significantly differ not only in dispersibility and morphology of individual particles [2, 11], but different mass macromolecules formed in the absence of regulatory documentation parameters characterizing the molecular weight distribution, and number-average molecular weight of the fractions with the highest specific gravity [2, 11], necessary to use special processing techniques to prepare the commercially available semi prior to processing.

Obviously, the most important aspect of the operation of preparation is approaching mass activity, the morphology of individual particles to optimize the values of the interparticle friction forces that have a significant effect on the kinetics of the process of cold pressing of the workpiece with a minimum of defects. Furthermore, it is necessary to ensure optimal mobility of macromolecules of different mass in the interparticle boundary layers formed during cold pressing, sintering the preform at the purpose of interdiffusion and formation of a monolithic structure. Obviously, for the implementation of this condition is an effective introduction to the interparticle boundary layer thermodynamically compatible modifiers in a controlled molecular weight distribution, acting as high-modifier.

On the supramolecular level of structure formation fluoro composite paradox manifested in the impossibility under the applicable process simultaneous production of a gradient structure with lamellar structure of the surface layer of the product provides a low shear resistance (low friction) in the operation of metal-supply system without external lubrication, and melkosferolitnym structure matrix in the amount of that raise the parameters of deformation and strength characteristics.

Processes of forming the optimal supramolecular structure in filled PTFE matrix during thermal exposure during mono politicization difficult due to the low mobility of macromolecules in the matrix binder. Therefore, an important role is played by the activity of the dispersed particles in the process of recrystallization of the original structure of the matrix of the PTFE particles. In the process of mixing the particulate filler particles are concentrated in the surface layer microroughnesses powdered binder particles that are able to supply industries have a significant range of size and morphological parameters.

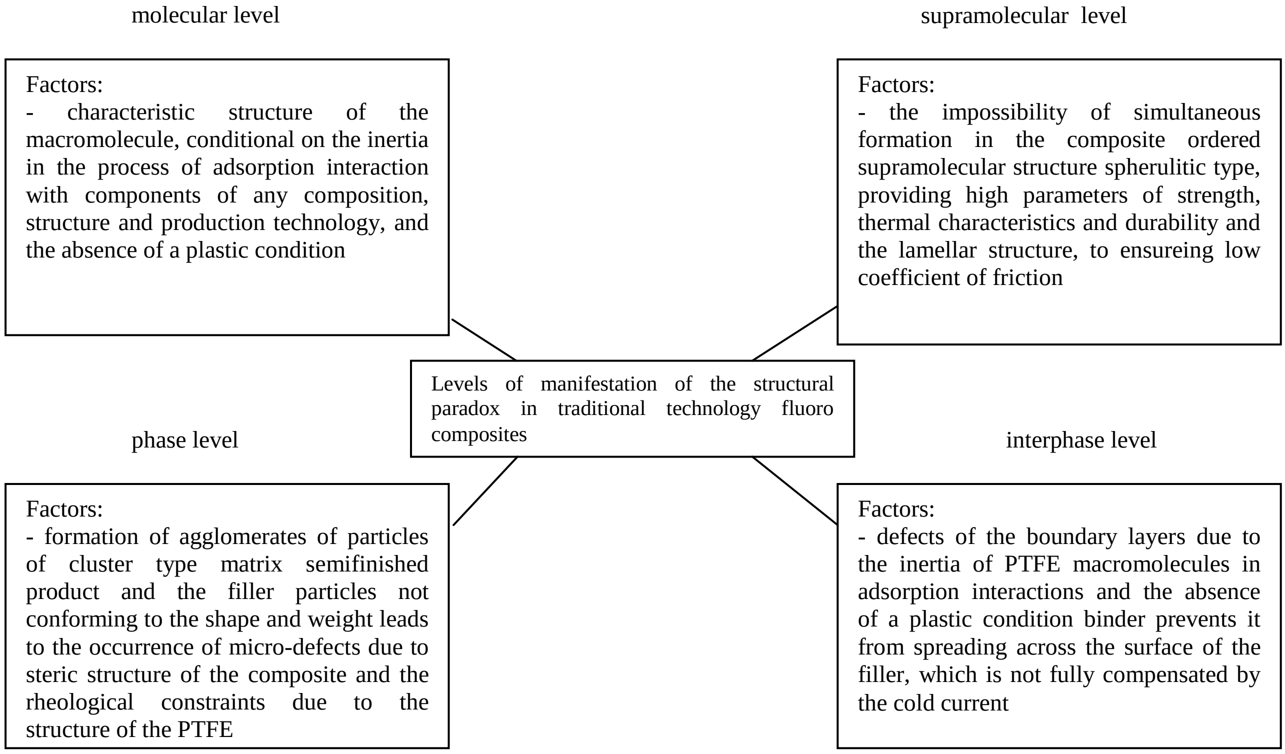

Fig. 2 *The main factors influencing the expression of the structural paradox in the formation fluorocomposites according to the technological paradigm*

This leads to the formation in step cold pressing the workpiece modifier concentration gradient and spatial grid formation, the shape and size of which depend primarily on the parameters of the particles in a state of an industrial semifinished product supply (Figure 4b). On stage mono politicization blank (hot sintering) dispersed particles modifier promote recrystallization of the matrix binder to form a supramolecular structure of a new type, size and morphological characteristics of which depend on the activity of the particles, the magnitude of its energy field, its impact on the mobility of macromolecules located in the periphery and their ability to spatial orientation to form ordered domains. Because of this factor to optimize the structure fluoro composite on supramolecular level sufficient doping [1, 3] content of dispersed components with sufficient activity in the range of values (0,001-1,0)% by weight. [1, 3, 6]. When an excess of the dispersed component in the composite formed cluster structures, impede the process of recrystallization of the matrix binder to form the optimal supramolecular structure and causing wear conjugated metal counterface. With the increase in the activity of single particles dispersed modifiers that can be achieved by using special technology education nanoscale fractions [1, 6], mechanical or thermal effect on the semi-finished product [1, 2, 4], the effectiveness of their actions on the supramolecular organization of the process increases, which provides modifying the desired effect at much lower concentrations [1, 7]. Therefore nanoscale modifiers (UDAG, zeolites, Sialons et al.), Subjected mechano-activated provide a pronounced effect of increasing the parameters of strength and tribological characteristics fluoro composites if they contain up to 1% by weight. [1, 4].

On the phase level education fluoro composite imperfect structure due to the formation in the mixing process of the particles of the matrix polymer and filler having a different shape, weight and electrical characteristics (Fig. 3a, b), the agglomerates of the cluster type (Fig. 3c), gives rise microdefects in the sample after cold pressing and remaining after sintering (mono politicization), due to the forces of interparticle friction and rheological steric obstacles for filling cavities connecting clusters (Fig. 3d).

Interphase level of imperfection structure of composite materials based on polytetrafluoroethylene, which leads to a decrease in the parameters of service characteristics, realized as a result of defects in the boundary layers in the "matrix-filler", leads to low strength, which is predetermined inert PTFE macromolecules in adsorption interaction and lack of bonding a plastic condition that prevents its spreading over the surface of the filler and increase the contribution of mechanical component into force of the adhesive interaction between the components. Therefore, in the composites filled with dispersed fragments of carbon, glass and other fibers, not realized their strength parameters, which in other thermoplastic matrices lead to a substantial increase in the parameter values and σ_t, σ_{comp}. For example, the parameter for σ_t and fiberglass reinforced plastics based on polyamide 6 (PA6) is from 110 to 130 MPa at a filler content of 20% by weight. The initial value of σ_t for the matrix polymer 60-65 MPa.

Pooled analysis shown in Figure 2 the main factors leading to the manifestation of the adverse effect of the parameters of strength and tribological characteristics fluoro composites with increasing filler content, indicates the dominant role of factors caused by peculiarities of technology of matrix binder (PTFE powder) and a reinforcing filler (HC) that aggravate the adverse effects characteristic of the chemical structure of the main components of the processes of forming the optimal structure at various levels of the organization by using traditional methods of mixing the components, cold extrusion billets and hot sintering (mono politicization). Therefore, to increase the parameter values of strength and wear resistance of the composites with a filler content of 20 wt.%,

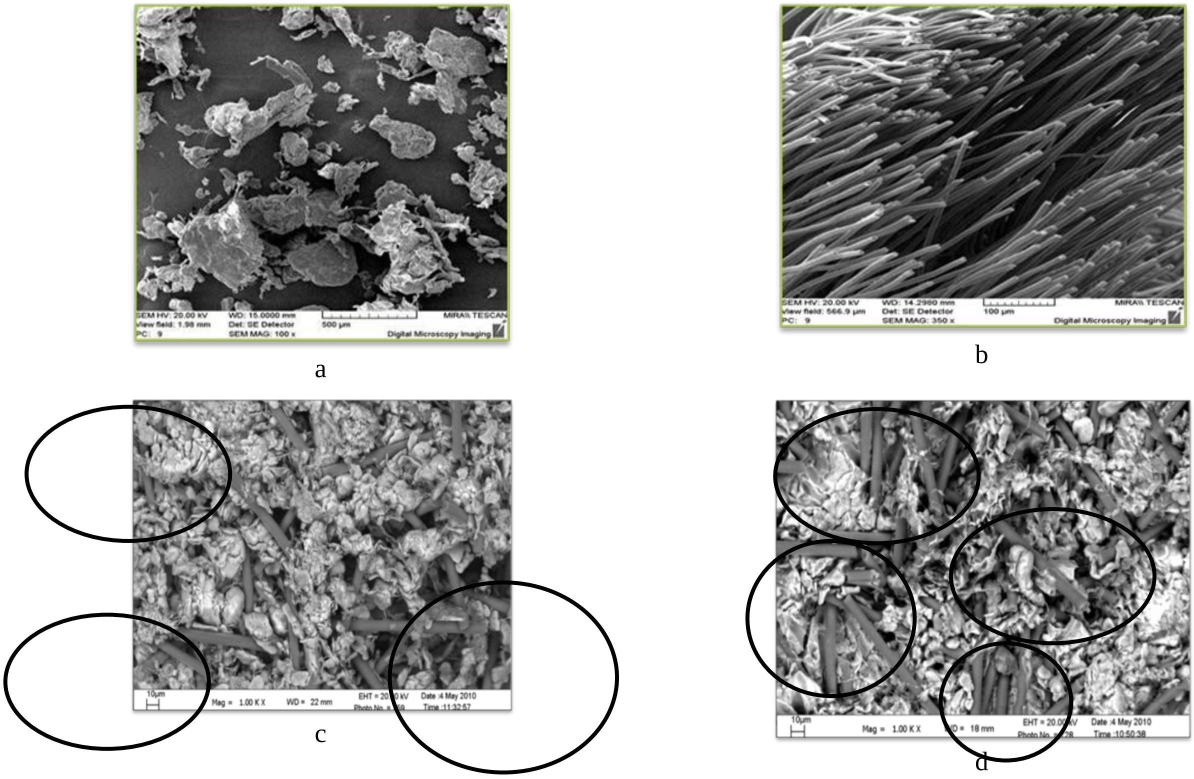

Fig. 3 *Characteristic form a matrix of polytetrafluoroethylene particles (a), of carbon fibers (b) and in the composition of the cluster structures of PTFE + 20% by weight. HC after mixing the components (c) and the product formed by the conventional technology (d). Clusters are marked by circles*

Related to the highly-fluoro composites, you must change traditional approaches technological paradigm, based on the features of the composition, structure and geometric parameters of the main components. The nature of these changes is as follows:

- it is necessary to carry out technological preparation components allow optimum parameters of geometrical characteristics of the dominant faction and reduce the negative impact of unstable molecular weight distribution of the polymer matrix on the parameters of strength, rheological properties and interparticle interaction;

- it is necessary to apply the technology of combining the components of the material to ensure destruction of the cluster structure formed by PTFE particles and hydrocarbons in the mixing process (Fig. 3, c);

- morphology characteristic matrix PTFE particles (Fig. 3, a) makes possible use as modifiers target components with high thermodynamic compatibility, which increases the ductility of the matrix in cold pressing and plasticizing effect due mono politicization. Effective modifier of this type are the synthetic products thermal gas dynamic polytetrafluoroethylene (UPTFE) containing oligomeric and polymeric fractions with similar molecular structure of the PTFE [6, 7];

- filling cavities cluster structures formed PTFE particles and hydrocarbons in the mixing process is possible through the use of fine particles UPTFE during the pre-modification tape semi-finished carbon fiber;

- cluster structure of the composite material is formed at the stage of mixing the components using a blade installations, allows it to implement the principle of multi-level modification of nanoscale components and micro-symmetric fractions with different forms of individual particles (carbon black, UPTFE, CF);

- when used as a modifier PTFE multi-dispersed carbon fiber role of morphological factors contributing to the possible formation of a concentration gradient of components of a heterogeneous system "PTFE-CF" prevails over the role of the intensity of the adsorption interaction at the interface "matrix modifier" providing education boundary layers optimum structure . Increased activity of the surface layer of hydrocarbons in interactions with PTFE by various technological factors (etching, oxidation treatment in the medium fluorinated gases and liquids, and other chemical plasma processing. [3, 6-8]) to form an interfacial layer of increased strength while maintaining the traditional technological receptions leveled the negative effect of defects formed by clusters of fragments of HC. Necessary to use technological methods, reduces the probability of formation of cluster structures in the composite modifier on stages of preparation components, their combination, pressing and mono politicization blanks.

Based on the above concepts developed efficient processing methods of forming composites little defect fluorinated structure with a filler content of 25-35 wt.%, Which belong to the class of highly filled (Fig. 4).

Practical implementation of the developed approach will allow to change the activity of the surface layer of hydrocarbon fragments in interactions with the matrix due to the consolidation of particles UPTFE, fill in the blanks clusters of fragments of CF components thermodynamically compatible with the matrix polymer, to bring the geometric shape and size of the PTFE particles and CF, reduce the likelihood of formation of clusters, the step of mixing the components in a paddle mixer, to destroy the structure of the cluster in the volume of the composite material during sintering (mono politicization) preform and its subsequent processing by plastic deformation. Obtained highly filled composites little defective structure, surpassing its analogues in the homogeneity and the parameters of strength and tribological characteristics (Table 1, Figure 1).

It should be emphasized that the developed methodological approaches are effective in obtaining not only fluoro composites containing carbon fiber (CF), and basalt, glass, etc. oksalonovoe. Kinds of heat-resistant high-strength fibers or mixtures thereof [12].

Thus, our system of research, including materials science, structural and technological aspects, allowed the development of highly efficient technology of composite materials based on polytetrafluoroethylene, exceeding the parameters of strength and tribological characteristics of the best industrial analogues "Fluvis" and "Flubon" [6, 14 -16].

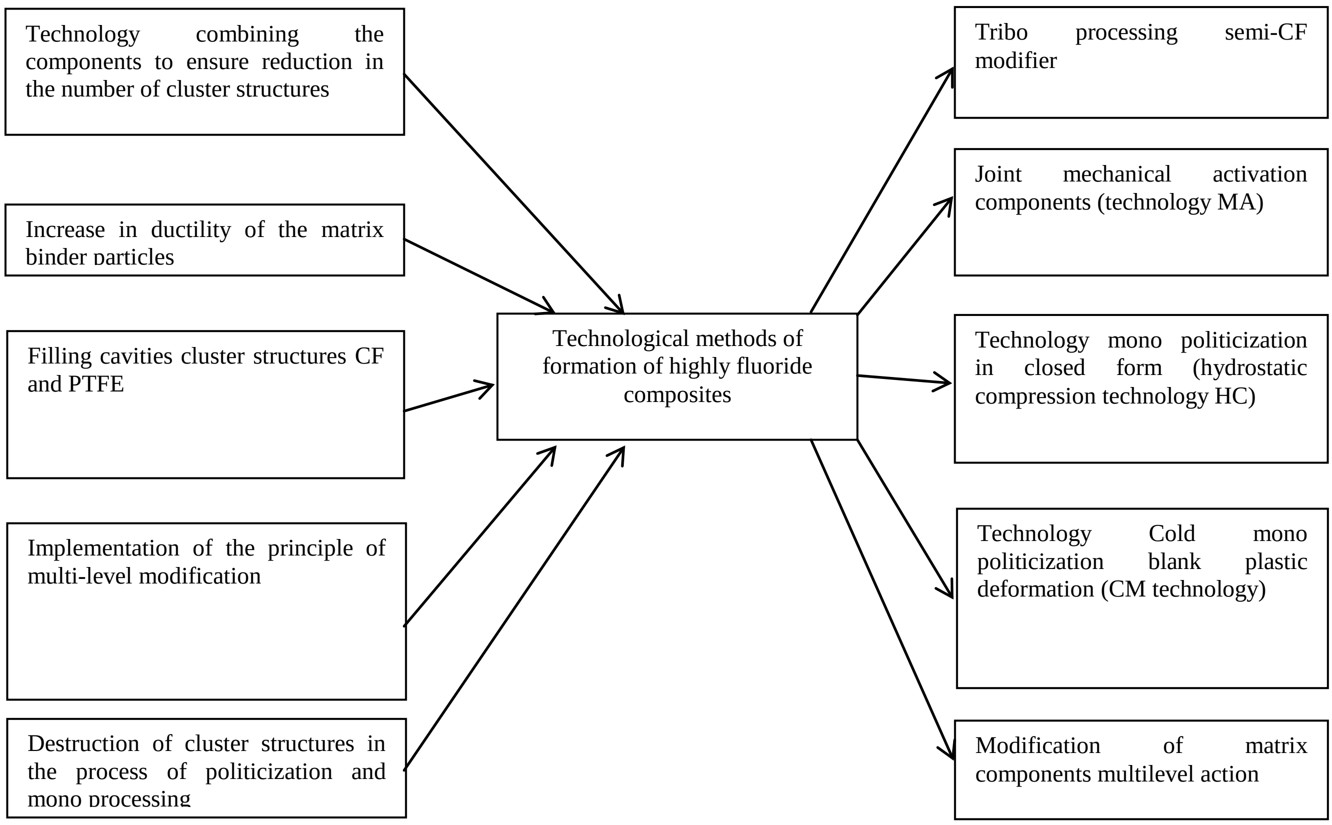

Fig. 4 *Technological methods of producing high-strength wear-resistant fluoro composites with a high content of carbon filler (CF)*

4. Conclusions

On the basis of studies:

- found that the main factor responsible for the manifestation of the structural paradox in traditional technology fluoride composites consisting in a proportional decrease in the values of parameters of strength and tribological characteristics with increasing degree of filling of the reinforcing filler is a discrepancy between the geometrical, dimensional and tribological parameters of the matrix particles (PTFE) and filler, leading to the formation of cluster structures in preparation of the composite, which is a prerequisite for the formation of macrodefects stages of cold pressing and subsequent sintering (mono politicization) products (billets);

- proposed effective technological methods to eliminate the structural paradox in highly composite materials based on polytetrafluoroethylene with a filler content of 20 wt.%, Based on the principle of multi-level modification implemented by the introduction of multi-component (UPTFE) [16] and reduce defects in the structure due to the destruction of clusters under the filler the influence of mechanical activation processes (technology MA), the thermal expansion of the components (technology HC) and the plastic flow of the matrix (CM technology) [13-15];

- The compositions of composite materials based on polytetrafluoroethylene, containing 25-35% by weight. reinforcing carbon fiber exceeding 1.5-2.0 times better analogy series "Flubon" and "Fluvis" the parameters of strength and tribological characteristics.

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