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Nanocomposite Polymer Materials with High Performance Characteristics

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Abstract. The practical implementation of methodological approaches to the creation of nanocomposite materials for various functional purposes based on industrial thermoplastics is considered. The concept of energy and technological compliance of components is proposed, which makes it possible to provide synergistic effects in improving the parameters of the performance characteristics of nanocomposites based on thermoplastic matrices. An algorithm for the formation of a multilevel optimized structure of nanocomposites based on thermoplastics with high melt viscosity has been developed. Compositions of functional nanocomposites for the manufacture of elements with high parameters of service characteristics, the priority and novelty of which are protected by patents for inventions, are presented.

Keywords: nanocomposite materials, nanomodifier content, formation algorithm, multilevel structure, concept of energy and technological compliance, compatibilization.

Introduction

A defining feature of the development of modern mechanical engineering, chemical industry, construction industry and power engineering is wide application of composites based on high-molecular matrices as structural, tribological and sealing materials. The range of polymer functional engineering composite materials is extremely broad and permanently expanding due to the emergence of industrial products synthesized from macromolecular compounds with enhanced performance characteristics, the development of highly effective technologies for targeted filler modification, and the creation of new types of modifiers and fillers. A special place among engineering materials based on polymer binders is occupied by a new class of composite materials, in the composition of which particles with the maximum size not more than 100 nm are introduced as a functional component. Such materials are commonly referred to as nanocomposite materials and distinguish them from traditional engineering materials due to their special properties caused by the active action of a low-dimensional modifier on the structure of the polymer matrix [1-5].

The properties of polymer nanocomposite materials depend on the composition of the components and the technology used to form the material and its product. Material science and technology of polymer nanocomposite materials are largely based on traditional approaches that take into account classical ideas about the mechanisms and kinetics of interphase processes in the system "matrix-functional filler". However, in some cases experimental data cannot be described by such approaches, which do not take into account the special energy state and morphology of nanoscale particles. In this regard, comprehensive research on the physicochemical, materials science, and technological aspects of the formation and processing of composite materials with low-dimensional modifiers is necessary to develop the foundations for creating unified methodological approaches that can reliably predict the parameters of their performance characteristics, as discussed in our previous study [6].

The aim of this research is the practical implementation of methodological approaches to the creation of nanocomposite materials for various functional applications based on industrial thermoplastics.

1. Research methodology

Thermoplastic polymers and oligomers, the most common in materials science and technology of polymeric materials, were used as components for obtaining composite materials for various purposes: aliphatic polyamides (PA6 and PA6.6, produced by Branch "Khimvolokno Plant" of JSC "Grodno Azot", Belarus; PA11 (Rilsan), produced by Arkema, France), polyolefins (PP, LDPE, HDPE, produced by JSC "Polimir", Belarus), fluorine-containing compounds (polytetrafluoroethylene (PTFE) F4 and F4-M (JSC "Halogen", Russia), products of thermogasodynamic synthesis of PTFE (ultradisperse polytetrafluoroethylene UPTFE), produced by Institute of Chemistry of the Far East Branch of the Russian Academy of Sciences, Russia.

Dispersed, including nanoscale, particles of carbon-containing (carbon nanotubes (CNT), carbon fibers (CF), ultradisperse diamonds (UDA), technical carbon (TC), silicon-containing (clays) and metal-containing (Cu formates) compounds obtained by technological effects on natural and synthetic semi-finished products produced at industrial enterprises of Belarus and the Russian Federation were used to control the structure parameters and performance characteristics of composites and products made of them.

The parameters of the stress-strain characteristics of the developed materials were evaluated on standard samples according to the relevant Russian standards. Tribological characteristics were determined on universal or original friction machines according to the schemes "indenter – disk", "shaft – partial liner". Assessment of the performance of products made of developed nanomaterials in the structures of automotive units for various purposes and technological equipment was carried out on stands and in the process of virtual tests using the SKIF supercomputer and full-scale tests.

2. Results and discussion

When choosing modifiers for the production of nanocomposite materials with optimized structure that determine the expediency and efficiency of their use in systems for a given functional purpose, *the principle of energy and technological compliance of components* was used, taking into account the concept of reasonable sufficiency and technological, environmental and economic aspects. The practical implementation of the developed methodological approaches ensures the achievement of technically significant effects of increasing the parameters of stress-strain, adhesive, tribological characteristics of composites based on industrial matrices both at the doping content (0.001-1.0 wt. %) of modifiers and at their content of 20–40 wt. %. At the same time, depending on the features of the composition, structure, dispersion and shape of the modifying particles, different levels of structural organization are realized. For highly dispersed particles at a doping content (0.001-1.0 wt. %) the structure is mainly optimized at the supramolecular and intermolecular levels [7–10]; when modifying matrices with active micrometer fragments ($80-150 \mu$ m) of high-modulus fibers (carbon, oxaloon, glass) at a content of 5–40 wt. % the prevailing role is played by the interfacial level [11, 12]. At the same time, modifiers can save the stability of the initial parameters of characteristics in the process of composite formation and operation of the product or metal-polymer system, or show lability due to transformation under the influence of physical and chemical processes, changing the initial structural parameters of the composite.

The mechanism of structuring action of nanosized particles (NSP) in polymer and oligomeric matrices of various structures is investigated. Based on the developed model, which assumes the formation of spherical supramolecular structures under the action of the active centers of the nanoparticle, an analytical expression was obtained to calculate the concentration of the modifier sufficient to transfer the entire matrix to an ordered state. As follows from [13, 14], the matrix will be completely modified provided that the modifier particle is active in the boundary layer with a thickness of $L = r [1 + (\rho_n/\rho_n)] ((1/C_n) - 1)^{1/3}$, where *r* is particle size; ρ_n , ρ_n are density of filler and polymer; C_n is filler content.

From this expression, it follows that the *L* is linearly related to the size of the nanoparticle and is practically independent of the ratio ρ_{μ} / ρ_n , that suggests similar mechanisms of modifying action in particles of different composition and production technology. Even with doping content (0.001–1.0 wt.%) the ratio of $1/C_{\mu} >> 1$ and the structuring effect of the nanoparticle on 2–3 adjacent layers of macromolecules provides a significant modification effect. Experimental studies have confirmed the adequacy of this conclusion (Fig. 1).

It has been established that the effectiveness of the modifying effect of a nanosized particle (NSP) is influenced not only by its size, composition and structure, but also by its shape. From an analytical expression that defines the size parameter of a nanoparticle characterized by a special energy state that affects the efficiency of the modifying action [15], $L_n = h \sqrt{3} \cdot \theta_D^{-1/2} / \sqrt{2m_e k}$, where *h*, *k* are Planck and Boltzmann constants, respectively, m_e is mass of electron, θ_D is Debye temperature, follows its dependence on the crystal-chemical direction. Therefore,

using modifier particles with relatively large sizes, the surface layer of which contains nanoscale components that meet the calculated value of L_n , it is possible to achieve technically significant modifying effects using available technologies for obtaining and processing polymer composites.

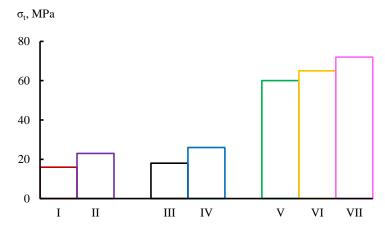


Fig. 1. – Tensile strength parameter σ_t for HDPE (I, II), PTFE (III, IV), PA 6 (V, VI, VII) initial (I, III, V) and modified by 0,05 wt. % UDD (II, VII), Cu (VI) and 0,5 wt. % UPTFE (IV)

Model concepts of the mechanisms of modification of high-molecular matrices by nanosized particles of various shapes have been developed. It is shown that when using particles of lamellar (scaly), whisker (fiber-like) and spherical shape, the degree of modification of the matrix M changes, determined by the ratio of the total modified volume to the total volume of the composite. Taking into account the different energy state of the NSP determined by the L_n parameter, and taking into account the anisotropic nature of the Debye temperature θ_D , the ratio of the degree of modification by scaly (M_{sc}) , whisker (M_{wh}) and spherical (M_{sph}) particles with the same content in the composite was obtained: $M_{sc}: M_{wh}: M_{sph} = 1: 0, 4: 0, 8$.

It follows from the obtained expression that when creating poorly filled nanocomposites based on polymer matrices, it is preferable to use layered modifiers, which include natural silicates, such as clays, micas, talc, which, under certain conditions, are able to disperse to form nanosized particles of a lamellar form [7-10].

The conducted experimental and theoretical studies are based on the assumption of invariability of the structure, composition and shape of the NSP introduced into the polymer matrix. At the same time, there are classes of NSP (metallic, oxide, metal-containing), which are able to transform as a result of physical and chemical processes occurring in the boundary layers of composites under the action of operational factors (thermal, mechanical, mechanochemical, etc.) with the formation of products of a different composition and structure and with a different mechanism of structurizing effect on the matrix polymer. Therefore, the justified choice of NSP and technologies for their production for the targeted modification of a polymer or oligomeric matrix involves a system analysis of structural-phase transformations, taking into account the energy and physicochemical aspects of the formation and functioning of a metal-polymer system within the framework of the concept of synergistic structuring based on the proposed methodological principles for the implementation of the nanostate phenomenon. The developed model concepts on the effect of energy and structural parameters of NSP on the efficiency of polymer matrix modification [7-15] made it possible to determine technological approaches to obtaining effective nanomodifiers using available semi-finished products in the form of layered minerals (clay, talc, mica, tripoli, etc.), as well as technological waste from chemical industries (products of refining vegetable oils, phosphogypsum, products of metallurgical production). The essence of the developed methods for obtaining silicate-containing NSP consists in the thermal effect on the dispersed particles of the semi-finished product obtained by dispersion in impact devices (grinders) with a temperature gradient of 800-1000 K in an air medium or an oxidation-free gas flow with a density $3 \cdot 10^6 - 8 \cdot 10^7$ W/m². Implementation of the developed methods for modification of polymer matrices of NSP in the amount of 0.1–10.0 wt. % or diffusion treatment of products made of polymer composites based on industrial thermoplastics (aliphatic polyamides PA6, PA6.6, PA11, polyolefins PP, HDPE) or their blends makes it possible to increase the strength parameters by 1.1-1.3 times, wear resistance by 1.5-2.0 times, as well as resistance to thermaloxidative environments due to multi-level modification.

The developed principles of multi-level inoculation of composite materials, the formation of an integrated supramolecular structure by modifying the matrix by a set of nanosized particles through catalysis of interfacial interaction make it possible to implement the concept of synergistic structuring in the production of nanocomposites based on thermoplastics, including those high melt viscosity (PTFE, UHMWPE). An algorithm has been developed for the formation of compositions of high-strength wear-resistant composites based on high melt viscosity matrices (PTFE, UHMWPE), methods of their manufacture, which provides an increase in the parameters of stress-strain characteristics in comparison with analogues (Fig. 2).

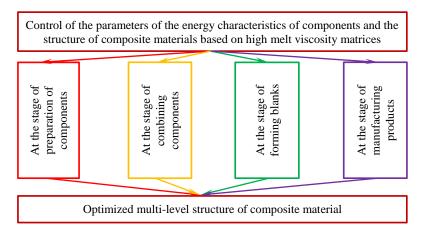


Fig. 2. – Algorithm for the formation of a multilevel optimized structure of composite materials based on thermoplastic matrices with high melt viscosity

When using nanoscale modifiers (carbon black, UPTFE, clay, talc), it is advisable to activate the components at the stages of preparation and combination using energy influences (thermal, laser). This approach has been tested in the development of technologies for the production of low-fill (0.5-5.0 wt%) nanocomposites using a combination of NSP (UPTFE, carbon black) and products of activation of layered minerals by thermal action in the temperature range 473–1373 K.

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For the development of low-fill composites based on high-viscosity matrices (UHMWPE, PTFE), options for implementing the principle of multilevel modification based on a combination of components with different resistance to transformation under the influence of technological factors are proposed. Within the framework of the current technological paradigm of fluorocomposites, an imperfect structure is formed due to the absence of a pronounced viscous-fluid state of PTFE, which prevents the processes of monolithization and interfacial interaction. The proposed combination of carbon-containing particles (CNT, carbon black) and UPTFE provides a reduction in structural heterogeneity due to the polymer-oligomeric structure and special rheological characteristics, which has a favorable effect on the performance characteristics of composite (Fig. 3). When used as modifiers of dispersed particles of thermoplastics (polyamides, polyolefins, polysulfone) under the influence of molding temperatures (573–623 K) for 8–24 hours, conditions are created for the formation of carbon-containing components with high thermodynamic compatibility with PTFE, with a shape adapted to interparticle defects, and high parameters of stress-strain characteristics ($\sigma_t = 85-90$ MPa). As a result of the transformation of the initial structure of the modifier, a composite is formed with a range of characteristics close to the characteristics of composites containing carbon fiber (CF) [16].

An effective modifier of the transformable type is the dispersed particles of oligomers selected from the oligoimide group (oligomaleidoaminophenylene, oligomaleidohydroxyphenylene, tetramaleimide, N, N'-bis-maleimides of unsaturated dicarboxylic acids, etc.). Carbonization processes under the influence of technological factors lead to the formation of carbon-containing particles with a nanoscale relief of the surface layer and increased activity in the processes of interfacial interaction, as a result of which structures with high parameters of stress-strain ($\sigma_t = 36-46$ MPa) and tribological ($I \times 10^7 = 0.5-2.0 \text{ mm}^3/\text{N} \cdot \text{m}$) characteristics are formed.

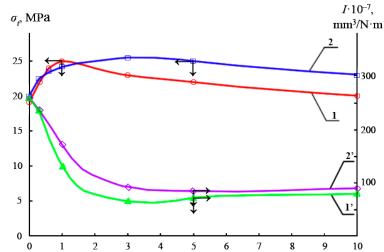


Fig. 3. – Dependence of tensile strength σ_t (1, 2) and wear intensity *I* (1', 2') of composite materials based on polytetrafluoroethylene modified with ultrafine polytetrafluoroethylene (UPTFE) on the content of dispersed particles of carbon black TU P324 (1, 1') and TU P803 (2, 2'). The content of UPTFE in composites is 2.0 wt. %

When developing composites with a filler with a size range of $50-150 \mu m$ and a content of 10-35 wt. % nanostate phenomenon can be realized within the framework of the concept of energy and technological compliance of components using mechanochemical activation.

Using the ideas about the mechanisms of structure formation of fluorocomposites formed using mechanically activated components developed by the scientific school of Prof. Okhlopkova A.A., methodological principles of fluorocomposite technology are proposed, in which fragments of carbon fiber (CF) are used as a modifier (Fig. 4).

Activation of components of composites based on PTFE, UHMWPE at the stage of forming blanks in accordance with patented technologies using common equipment for cold formation (LTF) with subsequent sintering made it possible to increase the tensile strength parameter for materials with a hydrocarbon content of 1-20 wt. % from 17-18 MPa to 20-35 MPa due to mechanochemical interaction at the filler-matrix interface in view of differences in thermal expansion coefficients.

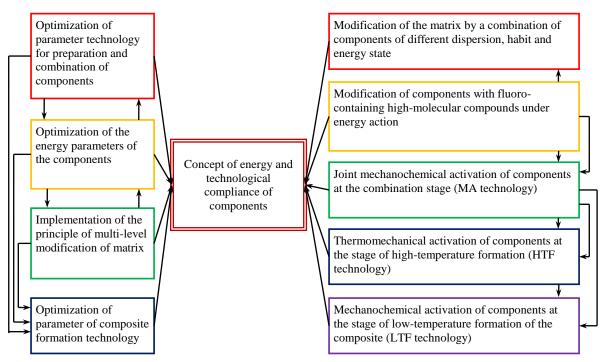


Fig. 4. - Methodological principles of technology of highly filled fluorocomposites with high performance parameters [17]

The proposed methods for achieving the nanostate of components using mechanical activation at the stages of combination (MA), high-temperature formation (HTF) and low-temperature formation (LTF) made it possible to develop a range of fluorocomposites with high performance parameters for the manufacture of elements of metal-polymer systems operated under the influence of elevated temperatures, absence or limitation of lubrication, reverse movement, exposure to active media, protected by patents of the Republic of Belarus for inventions. The combination of MA and HTF, MA and LTF technologies provides an opportunity to reduce the negative impact of the structural paradox noted in the works of A.K. Pugachev and Y.K. Mashkov (Fig. 5). The parameters of the characteristics of fluorocomposites obtained according to the proposed methodological principles in comparison with analogues are presented in [18–23]. As follows from Table 1, the developed composite compositions are superior in the main parameters of characteristics to the domestic and foreign analogues used (F4K20, Fluvis, Flubon) due to the implementation of the synergistic structuring effect, which made it possible to increase the carbon fiber content to 40–45 wt. % to increase the load characteristics of products made of such fluorocomposites.

	Parameter value for composite									
Characteristic	Flubon	Fluvis		Superfluvis		Composites obtained using developed technologies				
						Patent BY 9396	Patent BY 8480	Patent BY 9819		
Tensile stress, MPa	9	14	17*	18	27*	22–33	32–35	18–30		
Wear intensity at friction without lubrication, $I \times 10^{-7}$, 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		
Filler content, wt. %	30	30	20*	30	20*	20	20-40	20-45		

Table 1. Comparative performance parameters of PTFE-based composite materials

*Data of regulatory technical documentation for materials "Fluvis", "Superfluvis"

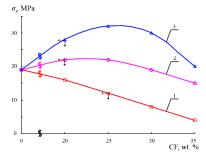


Fig. 5. – Dependence of tensile strength of PTFE-based composite materials on carbon fiber (CF) content in mechanical combination of components (1), in mechanochemical activation of components (MA) and formation in the free state (LTF) (2) and high-temperature formation (HTF) under conditions of triaxial compression (3)

Methodological principles for controlling the processes of structure formation of polymer matrices through the use of crystal-chemical parameters of nanosized particles of modifiers, the formation of an integrative supramolecular structure by modifying it with a set of nanoparticles of the same or different composition or shape are implemented in the development of composite materials based on industrial thermoplastics of the polyamide and polyolefin class.

The priority area of research was the development of composites for the manufacture of structural elements and the formation of multifunctional coatings used in the structures of automotive and tractor units (cardan shafts, brake chambers, shock absorbers) and technological equipment (belt conveyors) (Table 2).

The introduction of polyamide 11 into polyamide 6 in the presence of layered silicate nanoparticles provides an increase in adhesion and tribological characteristics [24]. The presence of nano-sized metal particles has a complex effect on the structure parameters, increasing the resistance of coatings to thermal-oxidative environments. The combined content of nanosized carbon-containing particles and fluorine-containing compounds contributes to the achievement of an optimal combination of adhesion, tribological characteristics and hydrophobicity of coatings, the parameters of which are considered in [25].

In the development of compositions of nanocomposite materials based on mixtures of polyamides, polyolefins (PA6, PA6.6, PA11, PP, HDPE, copolymer EVA), differing in thermodynamic compatibility, the established effect of physical compatibilization was used, due to the ability of nanosized particles to form intermolecular physical bonds of the adsorption type, experimentally confirmed by EPR spectroscopy and rheological studies.

	Parameter values for compositions							
Characteristic	PA6*	Composite material						
Characteristic		Patent	Patent BY 10898	Patent	Patent			
		BY 9397	Fatelit D 1 10090	BY 17434	BY 21059			
Tensile stress, MPa:								
– original	56-60	65–67	65–68	70–73	72–76			
– after 1,000 h of thermal oxidation at 423 K in air	18-30	-	58-62	43–45	—			
Adhesive strength (Falling indenter method), cm,	10-15	>50	45–51 MPa (Normal	28-30	50			
not less	10-15		tear-off method)	28-30				
Friction coefficient without lubrication at loads of	0.5-0.6	0.10-0.12		0,08-0,11	0,08-0,09			
5-10 MPa, $v = 0.5$ m/s	0.5-0.0	0.10-0.12	=	0,08–0,11	0,08-0,09			
Влагопоглощение, мас. %	9–10	-	_	3.0-3.5	2.0-3.0			

Table 2. Comparative characteristics of polyamide-based composite materials

*PA6 produced by JSC "Grodno Azot"

The compatibilizing effect of nanoparticles in the thermomechanical combination of components in a viscous state contributes to the formation of supramolecular and intermolecular hybrid structures. The function of a physical compatibilizer is characteristic of nano-sized particles of various composition, structure and production technology (clay, CNT, ultra-dispersed diamonds (UDD), Cu, etc.). This aspect made it possible to develop composite materials based on thermomechanically combined blends of polyamides and polyolefins with characteristics superior to analogues [26, 27].

Composites based on combined regenerated thermoplastic components are a full-fledged alternative to primary thermoplastics in the manufacture of engineering products for the construction industry (fasteners, metal-polymer rollers), including road construction (identification and restrictive elements for designating underground communications) in accordance with the developed regulatory and technical documentation – technical specifications TS BY 500037559.004-2021, TS BY 500037559.005-2021, TS BY 500037559.006-2021.

Comprehensive studies of the areas of practical implementation of the phenomenon of nanostate in materials science and technology of nanocomposites based on industrial thermoplastics have confirmed their validity and adequacy to the functioning industrial complex.

Conclusion

Based on the research, the following main results were obtained:

1) Methodological principles for the implementation of the nanostate phenomenon in materials science and technology of functional nanocomposites based on industrial thermoplastics of the polyolefin, polyamide, fluoroplastic class have been developed by optimizing the structure at the intermolecular, supramolecular and interfacial levels, ensuring the achievement of a synergistic combination of performance parameters. These principles are based on:

 – established crystal-chemical prerequisites for the selection of natural and synthetic carbon-containing, metal-containing and silicon-containing semi-finished products for the directed formation of active nanosized particles with specified structural, morphological and energy parameters under optimal technological action (mechanochemical, thermal, laser); implementation of the conditions for the energy compliance of nanomodifiers to the prevailing mechanism for the formation of the optimal structure of polymer, oligomeric and combined matrices at various levels of organization (molecular, supramolecular and interfacial);

- providing conditions for the manifestation of the preferred mechanisms of interfacial physicochemical interactions of components with the formation of boundary layers of optimal structure, which determine the mechanisms of destruction of nanocomposites under the influence of various operational factors.

2) Based on the concepts of condensed matter physics using the wave functions of the barrier model and the Debye-Waller factor, a theoretical analysis of the prerequisites for the formation of the nanostate of material objects in the form of dispersed particles and substrates in the process of their dispersion and technological impact on semi-finished products of various composition and structure is carried out. The mechanisms of formation of dispersed particles in the active state and charge mosaic on the surface of substrates under mechanical, mechanochemical and energy effects on semi-finished products are substantiated. An analytical substantiation of the change in the parameters of energy characteristics with a decrease in the geometric dimensions of a material object is given, confirming the hypothesis of the energy equivalent.

3) Physicochemical aspects of the modifying effect of nanoparticles of various composition and structure in thermoplastic matrices are considered. Characteristic features have been established that determine the activity of low-dimensional particles in the processes of transformation of the structure of the binder composite material at the molecular, intermolecular and supramolecular levels, consisting in their special energy state due to habit, size, factor and production technology. Using the proposed analytical expression, the analysis of the dimensional factor that determines the nanostate of the NSP of mono- and polyatomic substances, which determine their transition to a nanostate with high modifying activity in relation to high-molecular matrices, is carried out.

4) Features of the mechanism of modifying action of nanoparticles of various production technologies, composition and structure are considered. It is shown that the most important criterion of activity in the processes of adsorption interaction of a nanoparticle with macromolecules of a polymer or oligomeric matrix is its energy state, characterized by the presence of an uncompensated charge with a long relaxation time.

5) The methodological principle of multi-level modification is implemented in the creation of a range of nanocomposite functional materials based on industrial thermoplastics of the polyamide, polyolefin, fluoroplastic class with the introduction of a combination of components of different composition and dispersion with high energy characteristics. It is shown that the developed compositions based on aliphatic polyamides and polyolefins (PP, HDPE, LDPE) and their mixtures are superior to the base materials and their analogues by at least 1.2–1.5 times in terms of strength, wear resistance and adhesive characteristics. Fluorine-containing nanocomposites, semifinished products and products made of them, formed on the basis of the developed technological paradigm that ensures the formation of a low-defect structure due to the activation of interfacial interaction processes at the stages of combining components, pressing and formation, realize the synergistic effect of high parameters of stress-strain and tribological characteristics (σ_t , I) at least 1.5–2.6 times compared to domestic and foreign analogues formed using traditional technology.

6) Technologies for the implementation of the phenomenon of nanostate in the production of functional nanocomposite materials based on industrial thermoplastics and products from them for use in the structures of machines, mechanisms, technological equipment have been developed, providing the formation of an optimal multi-level structure to increase the service life. The technologies are based on the use of common equipment and are intended for sale at specialized enterprises for the industrial production of products from polymer and composite materials based on primary and regenerated raw materials.

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