

The practical applications of the concept of energy and technological compliance in materials science of polymer nanocomposites

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Abstract: The analysis of the mechanisms of implementation of the concept of multilevel modification in materials science and technology of polymer nanocomposites is carried out. It has been shown that the mechanisms of structure formation at the molecular, supramolecular and interfacial levels in nanocomposite materials based on industrial polymers of the class of polyamides, polyolefins, fluoroplastics modified by components that implement the nanostate phenomenon are based on:

– the formation of adsorption physical bonds in the boundary surface due to the energy interaction of active centers of nanosized particles with various shapes and components of the surface layer, which change the parameters of the rheological, stress-strain and adhesive characteristics of the composites;

– the demonstration of a structuring action by nanosized particles and components of the surface layer of particles, which manifests itself in the form of supramolecular and interphase ordering, physical compatibilization and non-chain stabilization, which increase the thermodynamic compatibility of the components of polymer-polymer and polymer-oligomer blends and inhibit thermo-oxidative and destructive processes in composites, causing a non-additive increase of the parameters of their stress-strain, adhesive and tribological characteristics.

KEYWORDS: NANOCOMPOSITES, POLYMER MATRICES, MULTILEVEL MODIFICATION, NANOSTATE, ENERGY AND TECHNOLOGICAL COMPLIANCE OF COMPONENTS

1. Introduction

Among the priority areas of the innovative strategy for the sustainable development of economic complexes of various levels is the practical implementation of the key component of convergent NBIC technologies – nanomaterials science and nanotechnology in order to increase the parameters of the characteristics of metal-polymer systems for various functional purposes.

In the nomenclature of modern engineering materials, a special place belongs to nanocomposites based on polymer, oligomeric and blend matrices, which, according to a number of parameters of functional characteristics, are uncontested materials in the production of motor transport, special, agricultural machinery, technological equipment, shut-off and control valves for heat power systems, petrochemical and processing industries. At the same time, an analysis of literary patent sources shows that the potential of industrial polymers in the creation of nanocomposites is not fully realized with a developed base of their large-tonnage production and equipment with modern technological equipment at the stages of obtaining and processing into products for various purposes. Therefore, the development of theoretical ideas about the mechanisms for the formation of the structure of nanocomposite materials based on industrially produced polymer and oligomeric matrices will make it possible to develop effective methodological approaches to the creation of domestic production of new generation composites in accordance with the needs of innovative enterprises.

The probability of the processes of formation of the structure of composite materials at different levels of organization is determined by the activation energy, which depends on the parameters of the energy characteristics of the components at a given technological impact. These parameters depend not only on the composition, but also on the dimensional and geometric features of material objects.

Studies of domestic and foreign experts have established the effect of the transition of a material particle upon reaching a certain size range into a state with special parameters of energy characteristics, called according to the established terminology a nanostate [1–4]. Obviously, the achievement of the nanostate by the component will have a significant impact on the structural processes in the composite material at different levels of organization, which determine the parameters of the stress-strain, tribological, adhesive and other characteristics of products in metal-polymer systems. At the same time, despite the experimentally established influence of energy parameters on structuring processes, there are no systemic studies of methodological approaches to the implementation of the nanostate phenomenon in materials science and the technology of nanocomposite materials based on industrial polymers. As a result, the achievement of synergistic effects of increasing the parameters of the performance characteristics of products from polymer

nanocomposites cannot be fully implemented in practical applications.

2. Methods of research

The main objects of study were nanodispersed particles of carbon-containing (graphite, DND, CNT, shungite, carbon fibers), metal-containing (oxides, salts of organic acids) and silicon-containing (mica, tripoli, opal, clay) compounds obtained by technological impacts on natural and synthetic semi-finished products produced at industrial enterprises in Belarus and the Russian Federation. Nanosized components were obtained by mechanical crushing and heat treatment of dispersed semi-finished products at temperatures of 673-1473 K.

Two main types of thermoplastic materials were used as polymer matrices. The first ones have hereditarily high viscosity (HHV) of the melt due to the chemical structure of the chain and molecular weight: polytetrafluoroethylene (PTFE) and super high-molecular polyethylene (SHMPE). The second group consisted of industrial thermoplastic polymers such as polyamide PA 6, high density polyethylene (HDPE), ethylene-vinyl acetate copolymers (EVAC), polypropylene (PP), thermoplastic polyurethane (TPU), etc. with characteristic parameters of rheological properties that changed upon the introduction of nanosized modifiers - acquired high viscosity (AHV).

Polymer materials were used in the state of industrial delivery in the form of granules or powder obtained by cryogenic dispersion of granules at a temperature of 87 K.

The structure and properties of nanocomposite materials and products produced from its were investigated using modern methods of physical and chemical analysis: IR spectroscopy (Specord), electron paramagnetic resonance (EPR) spectroscopy (RE 1306, Bruker), X-ray diffraction (Dron 2.0, Dron 3.0), differential thermal analyzes (Q-1500), optical (MIM-10, MF-2), scanning electron (ISM-50A, Nanolab-7) and atomic force (Nanotope III) microscopy. The energy state of nanoscale modifiers and composite materials was assessed from the EPR spectra and the spectra of thermally stimulated currents (TSC) on the original equipment of the V.A. Belyi Metal-Polymer Research Institute of National Academy of Sciences of Belarus (MPRI NASB). The dielectric characteristics of materials after energy exposure (laser, ion, temperature) were determined according to the appropriate standardized methods. The regulation of the nanorelief of the surface layer of polymer samples and fillers was carried out using a short-pulse laser and accelerated ion irradiation with a given power density.

The parameters of the stress-strain characteristics of the developed materials were evaluated on standard samples in accordance with the relevant standards (GOST). Tribological characteristics were

determined on universal or original friction machines (UMT, MI-2, SMC-2M, etc.) according to the "indenter – disk", "shaft – partial insert" schemes. Evaluation of the performance of products from the developed nanomaterials in the designs of automotive units for various purposes and technological equipment was carried out at the stands and in the process of virtual tests using the SKIF supercomputer and field tests. The test data processing was carried out by the methods of mathematical statistics using the standard software Microsoft Office 2020.

3. Results and discussion

When choosing modifiers for obtaining nanocomposite materials with optimized structural parameters that determine the feasibility and efficiency of their use in systems of a given functional purpose, we proceeded from the principle of energy and technological compliance of components based on the concept of reasonable sufficiency, taking into account technological, environmental and economic aspects [5–7]. Practical implementation of the developed methodological approaches ensures the achievement of technically significant effects of increasing the parameters of the stress-strain, adhesive, tribological characteristics of composites based on industrial matrices both with a doping content (0.001–1.0 wt. %) of modifiers and with its content of 20–40 wt. %. In this case, depending on the characteristics of the composition, structure, dispersion and shape of the modifying particles, various levels of structural organization are realized. For highly dispersed particles with a doping content (0.001–1.0 wt. %), the structure is mainly optimized at the supramolecular and intermolecular levels [8, 9]; when modifying matrices with active micrometer fragments (80–150 μm) of high modulus fibers (carbon, oxalone, glass) at a content of 5–40 wt. % the interfacial level plays a predominant role. In this case, modifiers can maintain the stability of the initial parameters of the characteristics during the formation of the composite and the operation of the product or metal-polymer system, or exhibit lability due to transformation under the influence of physicochemical processes, changing the initial structural parameters of the composite [10].

The mechanism of the structuring action of nanosized particles (NSP) in polymer and oligomeric matrices of various structures has been investigated. Based on the developed model, which assumes the formation of supramolecular spherical structures under the action of active centers of the nanoparticle, an analytical expression is obtained to calculate the concentration of the modifier sufficient to transfer the entire matrix to an ordered state.

As follows from [11, 12], the matrix will be completely modified provided that the modifier particle exhibits its activity in the boundary layer with thickness $L = r [1 + (\rho_f/\rho_p)] ((1/C_f) - 1)^{1/3}$, where r is the particle size; ρ_f, ρ_p – density of the filler and polymer; C_f – the content of the filler.

It follows from this expression that the parameter L is linearly related to the size of the nanoparticle and practically does not depend on the ratio ρ_f/ρ_p , which suggests similar mechanisms of the modifying action for particles of different composition and production technology. Even with a doping content (0.001–1.0 wt. %), the ratio $1/C_f \gg 1$ 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 was found that the efficiency of the modifying action of a nanosized particle (NSP) is influenced not only by the size, composition, and structure, but also by its shape. From the analytical expression that determines the size parameter of a nanoparticle, characterized by a special energy state that affects the efficiency of the modifying action, $L_n = h \sqrt{3} \cdot \theta_D^{-1/2} \sqrt{2m_e k}$, where h, k are the Planck and Boltzmann constants, respectively, m_e is the electron mass, θ_D is the Debye temperature, it follows its dependence on the crystal chemical direction [11, 12]. Therefore, using relatively large modifier particles, the surface layer of which contains nanosized components that satisfy the calculated L_n value,

it is possible to achieve technically significant modification effects using available technologies for the production and processing of polymer composites.

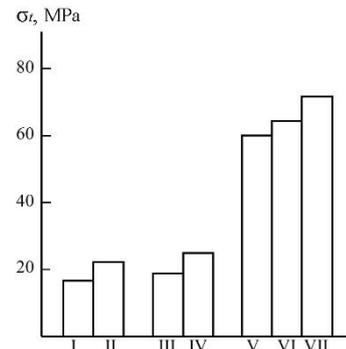


Fig. 1. Ultimate tensile strength σ_t for HDPE (I, II), PTFE (III, IV), PA 6 (V, VI, VII) of the original (I, III, V) and modified 0.05 wt. % DND (II, VII), Cu (VI) and 0.5 wt. % UPTFE (IV)

Model concepts of the mechanisms of modification of high-molecular-weight matrices by nanosized particles of various shapes have been developed. It is shown that the use of lamellar (scaly), whisker (fibrous) and spherical particles changes the degree of modification of the matrix M, which is 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 parameter L_n , 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 was obtained with the same content in the composite: $M_{sc} : M_{wh} : M_{sph} = 1 : 0,4 : 0,8$.

It follows from the obtained expression that when creating low-filled nanocomposites based on polymer matrices, it is preferable to use layered modifiers, which include natural silicates – clays, mica, talc, which, under certain conditions, are capable of dispersing with the formation of nanosized lamellar particles.

The carried out experimental and theoretical studies are based on the assumption that the structure, composition and shape of the NSP introduced into the polymer matrix remain unchanged. At the same time, there are classes of NSP (metal, oxide, metal-containing), which are capable of transforming as a result of physicochemical processes occurring in the boundary layers of composites under the action of operational factors (thermal, mechanical, mechanochemical, etc.) with the formation of products of another composition and structure with an excellent mechanism of structuring action on the matrix polymer. Therefore, a reasonable choice of NSP and technologies for its production for the implementation of targeted modification of a polymer or oligomeric matrix presupposes a systematic analysis of structural and phase transformations, taking into account the energetic 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 of the influence of the energy and structural parameters of NSP on the efficiency of modification of polymer matrices made it possible to determine technological approaches to obtaining effective nanomodifiers using available semi-finished products in the form of layered minerals (clays, talc, mica, tripoli, etc.), as well as technological waste of chemical production (refined products of vegetable oils, phosphogypsum, products of metallurgical industries). The essence of the developed methods for the preparation of silicate-containing NSP consists in the thermal effect on dispersed particles of a semi-finished product obtained by dispersing in kinetic impact machine (crushers) with a temperature gradient of 800–1000 K in air or in a non-oxidizing gas flow with a density of $3 \cdot 10^6$ – $8 \cdot 10^7$ W/m². Intense thermal action leads to the destruction of the initial particles with the formation of nanosized fractions of various structures and compositions [12].

Preliminary modification of semi-finished products of layered minerals (clays, talc) with thermally decomposable compounds such as carbonates, formates, metal oxalates, organic refining products of vegetable oils, water-soluble polyamides increases the intensity of degradation processes by the stratification mechanism with the formation of lamellar particles. The developed methods make it possible to obtain effective modifiers of industrial thermoplastics with a size range of 10–30 nm and a long-term active state.

Implementation of the developed methods when modifying polymer matrices of NSP in an amount of 0.1÷10.0 wt. % or diffusion treatment of products made of polymer composites based on industrial thermoplastics (aliphatic polyamides PA 6, PA 6.6, PA 11, polyolefins PP, HDPE) or its blends allows increasing the strength parameters by 1.1÷1.3 times, wear resistance by 1.5÷2.0 times, as well as resistance to the effects of thermo-oxidizing environments due to multilevel modification.

The developed principles of multilevel modification of composite materials, the formation of an integrated supramolecular structure by modifying the matrix with a set of nanosized particles by means of interfacial interaction catalysis make it possible to implement the concept of synergistic structuring in the preparation of nanocomposites based on thermoplastics, including those with increased melt viscosity (PTFE, SHMPE). An algorithm for the formation of compositions of high-strength wear-resistant composites based on high-viscosity matrices (PTFE, SHMPE), methods of their manufacture, providing an increase in the parameters of deformation-strength characteristics in comparison with analogs, has been developed (Fig. 2).

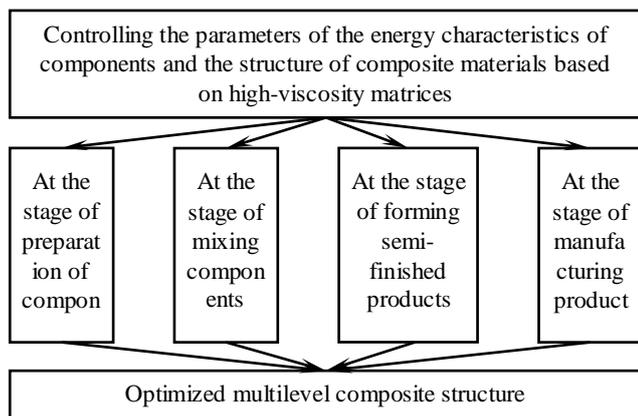


Fig. 2. Algorithm for the formation of a multilevel optimized structure of composite materials based on thermoplastic matrices with increased viscosity

When using nanosized modifiers (carbon black (CB), UPTFE, clay, talc), it is advisable to activate the components at the stages of preparation and mixing using energy (thermal, laser) impact. This approach was tested in the development of technologies for the production of low-filled (0.5÷5.0 wt.%) nanocomposites using a combination of NSP (UPTFE, CB) and products of the activation of layered minerals by thermal action in the temperature range 473–1373 K. For the development of low-filled composites based on high-viscosity matrices (SHMPE, 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, have been proposed. Within the framework of the existing technological paradigm of fluorine composites, an imperfect structure is formed due to the absence of a pronounced viscous-fluid state of PTFE, which prevents the processes of monolithization and interphase interaction. The proposed combination of carbon-containing particles (CNT, CB) and UPTFE provides, due to the polymer-oligomeric structure and

special rheological characteristics, a decrease in structural heterogeneity, which favorably affects the parameters of the composite characteristics (Fig. 3). When dispersed particles of thermoplastics (polyamides, polyolefins, polysulfone) are used as modifiers under the influence of molding temperatures (573–623 K) for 8–24 hours, conditions are created for the formation of carbon-containing components with increased thermodynamic compatibility with PTFE, with a shape adapted to interparticle defects, and high parameters of stress-strain characteristics ($\sigma_r = 85\div90$ MPa).

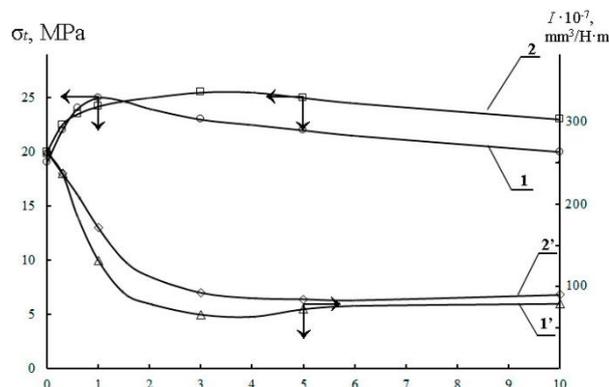


Fig. 3. Dependence of ultimate tensile strength (1, 2) and wear rate (1', 2') of composite materials based on polytetrafluoroethylene modified with ultrafine polytetrafluoroethylene (UPTFE) on the content of dispersed particles of carbon black CB P324 (1, 1') and CB P803 (2, 2'). The content of UPTFE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

Dispersed particles of oligomers selected from the group of oligoimides (oligomaleimidoaminophenylene, oligomaleimidohydroxyphenylene, tetramaleimide, N, N'-bis-maleimides of unsaturated dicarboxylic acids, etc.) are an effective modifier of the transformable type. 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 high activity in the processes of interphase interaction, as a result of which structures with increased parameters of stress-strain ($\sigma_r = 36\text{--}46$ MPa) and tribological ($I \times 10^7 = 0.5\text{--}2.0$ mm³/N·m) characteristics.

When developing composites with a filler with a size range of 50–150 μm and a content of 10–35 wt. % the phenomenon of nanostate can be realized within the framework of the concept of energy and technological correspondence using mechanochemical activation.

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

Activation of the components of composites based on PTFE, SHMPE at the stage of forming semi-finished products in accordance with patented technologies with the use of common equipment for cold monolithization followed by sintering made it possible to increase the ultimate tensile strength parameter for materials with CF content of 1–20 wt. % from 17–18 MPa to 20–35 MPa due to the provision of mechanochemical interaction at the “filler - matrix” interface due to the difference in the coefficients of thermal expansion.

The developed options for applications of the concept of energy and technological compliance are presented in table.

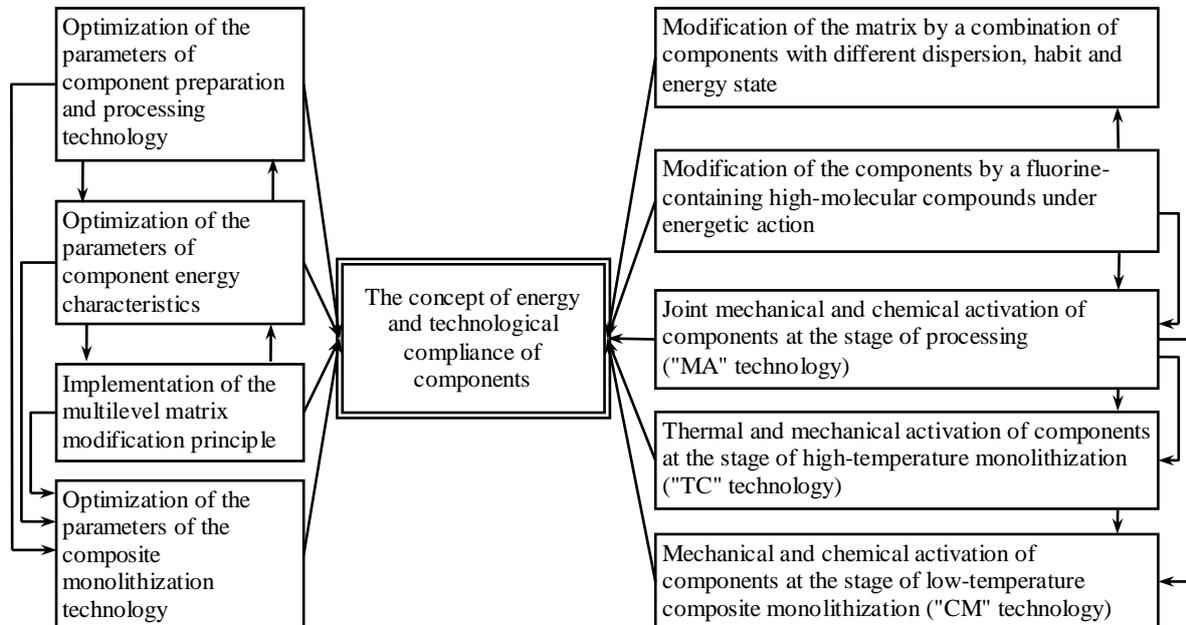


Fig. 4. Methodological principles of the technology of highly filled fluorine composites with increased performance parameters

Table. Practical applications of the concept of energy and technological compliance in materials science and technology of nanocomposites based on thermoplastics

Methodological principle of implementation	Developed material science and technological solution	Structural features of the nanocomposite and the achieved technical effects	The level of novelty of the developed solution
1	2	3	4
Physical compatibilization of blend components by nanoparticles	Introduction of nanosized particles under thermomechanical mixing of components	The formation of blend structure due to the formation of intermolecular bonds of the adsorption type. Increasing the parameters of stress-strain, tribological characteristics and resistance to thermal oxidative aging	BY patents for inventions No. 8674, 9215, 9820, 9397, 10585, 10986, 17434. RU patents for inventions No. 2265037, 2266988, 2268273, 2276677, 2278875, 2283325, 2305117, 2309964.
The principle of multilevel modification of the composite material matrix	Introduction of a set of modifying particles with different size parameters, energy activity and shape	Formation of an integrated structure with intermolecular, supramolecular and interfacial components. Increasing the parameters of stress-strain characteristics and wear resistance	BY patents for inventions No. 8480, 11421, 21061.
Mechanochemical activation of components at various stages of the technological process	Mechanochemical action on components in kinetic impact or friction-type machines at the stages of preparation, mixing of components, processing of products (semi-finished products)	Formation of products with nonstoichiometric composition due to intermolecular and interphase interaction of components, a decrease in the number of structural defects, an increase in thermodynamic compatibility. Increasing the parameters of the stress-strain characteristics of products	BY patents for inventions No. 8480, 9396, 14813, 17180, 17719, 18089, 21059, 21060, 21061, 21397, 21900. RU patents for inventions No. 2266925, 2266988.
The use of crystal chemical parameters of dispersed modifiers for structuring matrices at various levels of organization	Thermal, thermomechanical, laser, ionizing effects on a semi-finished product in the form of microparticles, fibers, films to obtain nanoscale fractions or nanorelief of the surface layer	Structuring a matrix with a high level of ordering at various levels of organization under the influence of the nanostate factor of disperse particles or relief components. Increasing the parameters of stress-strain, adhesive, tribological characteristics of products and coatings	BY patents for inventions No. 8999, 10391, 10795, 10901, 10898, 11382, 11694, 17248, 17719, 18063, 22316. RU patents for inventions No. 2269554, 2332524, 2332525, 2307855, 2321603, 2329285, 2338764. UZ patent for invention No. 06365.
Inhibition of the processes of corrosion and mechanical wear by separating layers of nanocomposite materials	Introduction to the friction contact zone of carbon-containing, silicate-containing particles in combination with oligomeric and polymer components and oils	Formation of a nanostructured separating layer in the contact interaction zone with low resistance to tangential shear and the ability to signal-alternating transfer	BY patents for inventions No. 7832, 8470, 18073. RU patents for inventions No. 2243998, 2248389.

1	2	3	4
Recovery of the structural potential of regenerated thermoplastics by doping modification with nanosized particles	Modification of regenerated thermoplastic matrices with a doping amount of nanosized particles (0.001–0.1 wt. %) in the process of thermomechanical action on the melt	Formation of a predominantly spherulite structure of the matrix polymer with the participation of radical fragments of aging and thermal mechanical destruction. Increasing the parameters of stress-strain characteristics and resistance to service life of products	BY patents for inventions No. 9820, 10586. BY patents for utility models No. 11240, 11241, 11264, 11265, 11266, 11630, 11631, 11634, 11635, 11773, 11776, 11882, 11967. RU patents for inventions No. 2283325, 2305117, 2309964. RU patent for utility model No. 170525.

The comprehensive studies of the directions of the practical implementation of the nanostate phenomenon in materials science and the technology of nanocomposites based on industrial thermoplastics have confirmed their validity and adequacy to a functioning industrial enterprises.

4. Conclusion

Methodological principles have been developed for the implementation of the nanostate phenomenon in material science and technology of functional nanocomposites based on industrial thermoplastics of the class of polyolefins, polyamides, fluoroplastics by optimizing the structure at the intermolecular, supramolecular and interfacial levels, ensuring the achievement of a synergistic combination of performance parameters 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 given structural, morphological and energy parameters under optimal technological (mechanochemical, thermal, laser) impact;
- the implementation of the conditions for the energy compliance of nanomodifiers with the dominant mechanism for the formation of the optimal structure of polymer, oligomer and blend matrices at various levels of organization – molecular, supramolecular and interfacial;
- ensuring the conditions for the manifestation of the preferred mechanisms of interphase physical and chemical interactions of components with the formation of boundary layers with optimal structure, which determine the mechanisms of destruction of nanocomposites under the influence of various operational factors.

The physical and chemical aspects of the modifying effect of nanoparticles of various compositions and structures in thermoplastic matrices are considered. The features that determine the activity of low-size 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 the habit, size of the factor and the production technology, have been established. Using the proposed analytical expression, the analysis of the size factor determining the nanostate of NSP of mono- and polyatomic substances, which determine its transition to the nanostate with increased modifying activity to high-molecular matrices, has been carried out.

The features of the mechanism of the 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 oligomer matrix is its energy state, characterized by the presence of an uncompensated charge with a long relaxation time.

Variants of practical application of the concept of energy and technological compliance in materials science and technology of nanocomposites based on industrial thermoplastics have been developed.

5. Acknowledgements

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