INFLUENCE OF POLYMER-SILICATE NUCLEATOR ON THE STRUCTURE AND PROPERTIES OF POLYAMIDE 6

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Abstract. Physico-chemical properties of developed nickel-containing modified silicate materials as the nucleators for composites based on polyamide 6 are presented. As it was established, additive of the nucleator modified by polyvinylpyrrolidone promotes the increase of crystallinity degree of polyamide 6 and the decrease of crystallite size. It was found that changes in the structure of the material cause the increase of its tensile strength, surface hardness and Vicat softening point.

Keywords: nucleator, polyvinylpyrrolidone, crystallization, polyamide 6, composite.

1. Introduction

Among the physical methods of polymer materials modification which allow directional influence on the supramolecular structure and properties the prevalent are: creation of polymer blends of different nature (thermoplastic with each other and thermosetting with thermoplastic [1-3]); creation of polymer composites with inert fillers of different nature [4-6]; modification of polymer materials with active additives (nucleators, plasticizers, foamers [7-9]).

Among the nucleation additives that can be used to influence the morphology of polymeric materials, fine metal-containing silicate materials have been widely used [10, 11]. These additives can directly affect the size of crystalline formations, the degree of crystallinity and the performance characteristics of polymeric materials.

In this case the morphology of polymers depends on many factors. These are configuration and conformation of their macromolecules, chemical composition of

elementary units and of macromolecule as a whole, size of individual atoms and structure of its orbitals, conditions under which the structuring took place, duration and velocity of structure formation, *etc.* [1, 5]. It should be noted that the supramolecular structure of the polymer is also affected by the conditions of its synthesis, storage and processing.

The study of supramolecular structure of polymers seems to be very important in terms of its interrelation with a complex of physical properties of material, velocity and mechanism of physico-chemical and chemical processes, as well phenomena during its processing. Owing to differences in supramolecular structure the products synthesized from the same polymer but under different conditions often have different properties.

Moreover, the structure of the polymer changes significantly during its processing and depends on the conditions under which it is carried out. As the result there is a different degree of the polymers crystallinity and the presence of interval in melting point of the crystals.

The degree of crystallinity of polymers and composites on its basis depends not only on the micro-rheological processes at the processing stage, but are mostly determined by the nature of the initial components [1], technological mixing parameters, interfacial interactions at the dividing border [12], the ability of the components to form the crystalline phase [13], the presence of various additives, *etc.* [14]. Therefore, the establishment of morphological features of the studied materials makes it possible not only to determine the factors affecting the technological compatibility of the components in given materials, but also to predict their technological and operational properties [15]. At the same time polyamide 6 (PA-6) is one of the most widely used thermoplastics. However, for some areas of application the increased physico-mechanical and thermophysical properties are required.

This paper presents the results of studies of fundamental physico-mechanical, elastic-deformation and thermophysical properties of polyamide composite materials with Ni-containing nucleating agent in relation to their supramolecular structure.

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2. Experimental

210/310 polyamide 6 (Grodno Khimvolokno OAO, Belarus), Ni-silicate nucleating agent (Ni-NA) and Ni-silicate nucleating agent modified by polyvinyl-pyrrolidone (Ni-PVP-NA) were used to obtain thermo-plastic composites based on PA-6. Ni-containing silicate nucleator was obtained by the action of a solution of nickel chloride on sodium liquid glass [16, 17]. In the case of NA modified by polyvinylpyrrolidone the PVP was previously dissolved in sodium liquid glass.

The components were pre-mixed with the desired ratio in a drum type mixer (powdering a granular thermoplastic with a nucleation agent) for 15–20 min. After obtaining the mechanical blend, it was dried for 8–10 h in an air vacuum dryer of 2B-131 type at 363 K. Further the homogenization of the blend was conducted *via* mixing components in a viscous state on a Cellier extruder, which is equipped with a screw of 700 mm length and 25 mm in diameter. The rotation velocity of the extruder screw was 10–15 rpm. The temperatures of the extruder zones were 453–463, 473–483, and 493–508 K. The obtained extrudate was crushed on a crusher gear type. The production of standard samples of granular material for research was conducted by injection molding on a Krauss Maffei KM 110-520 C2 molding machine. The filler content was 10 wt %.

To determine the sorption properties of nucleator, the photocolorimeter KFK-2 with methylene blue as an indicator were applied. The material was stirred with the indicator at 298 ± 1 K for 1 h, then the solution was centrifuged, and the optical density was measured.

To determine the effect of nucleating agent on supramolecular structure of polyamide 6 the X-ray diffraction studies were carried out applying a DRON-4-07 diffractometer with a Cu-anode lamp and a Ni filter. The results of the studies were processed using the WAXSFIT software [18], by means of which an approximation of the experimental diffraction curve was carried out which corresponds to the change in the X-ray scattering intensity (imp/s) depending on the angle of diffraction $2\theta,°$. The approximation was conducted following Savitsky-Golay method. Application of this software allowed to determine the position of crystalline peaks, as well as to optimize the process of decomposition of the diffraction curve into components.

Researches of the elastic-deformational properties and the structure coefficient of the obtained materials were carried out using a Hepler consistometer at 293 K by indentation of a cone-shaped indenter under the load of 120 N. The characteristics were determined by the modulus-deformation method of calculation [19].

The coefficient of linear thermal expansion of the investigated materials was determined according to ISO 11359.

The SPECORD 70 spectrograph was applied for FTIR spectroscopy; the spectra were recorded within the range of 400–4000 cm^{-1}. Samples were prepared by pressing tablets with KBr.

SEM studies were performed applying a REMMA 102-02 scanning electron microscope. The sample surface was scanned using an electron beam with a diameter of several nanometers and an electron energy of 0.2–40 kV. Zoom change range was 10–30000, the resolution was about 5 nm. Researches were conducted on material with a conductive coating.

Vicat softening point of the investigated materials was determined in accordance with ISO 306: 2013B, the loading was 50 N.

The surface hardness of the conical fluidity point was determined applying Hepler Consistometer at 293 K by indentation of the steel zone in the polymer sample with a sharpening angle of 58°08' under the load of 50 N for 60 s.

Tensile strength σ_{ts} and tensile elongation ε_{ts} were determined according to ISO 527-1, -2, using TIRA Test 2200 tensile testing machines. The method is based on stretching the test specimen with a specified strain rate. Standard samples were used for the study. The strain rate was 5 mm/min.

All experimental results of physico-mechanical and thermophysical properties studies are the average values of at least 3 experiments.

3. Results and Discussion

One of the technological methods of influencing the morphology and properties of PA-6 is the introduction of additives of various physical action into the products during the processing, in particular nucleating agents, which are able to change the regularities of the crystallization, the crystallinity degree and the size of the polymer crystallites [20, 21]. Among the nucleating agents the nanosized particles of inorganic nature, in particular modified silicates, are widely applied [22, 23].

In presented work, pre-synthesized nickel-containing silicate materials were used as nucleators for polyamide 6. These materials were obtained as a result of the co-sedimentation of sodium liquid glass and functionally active modifier – polyvinylpyrrolidone under the action of nickel chloride. The obtained materials are characterized by a variety of active surface groups:
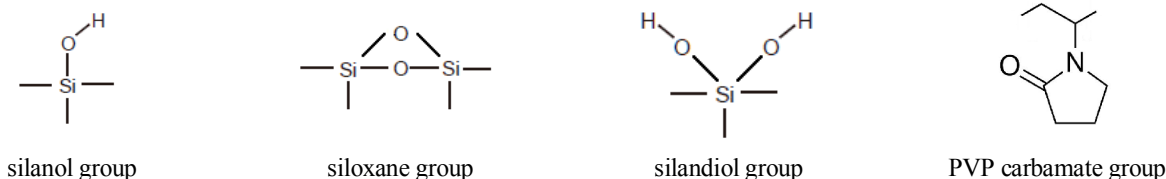
silanol group   siloxane group   silandiol group   PVP carbamate group

Table 1

Elemental composition and sorption properties of nickel-containing nucleating agent

Nucleating agent	Element			Ratio		Number of active centers $q_a \cdot 10^6$, mol/g	Specific area of the active surface S_a, m²/g
	O	Si	Ni	Ni/Si-O	O/Si		
	Content, %						
Ni-NA	57.16	19.58	23.26	0.30	2.45	99.10	77.58
Ni-PVP-NA	63.33	17.78	18.89	0.23	3.56	94.15	73.70

An increase in the oxygen:silicon ratio in the modified nucleating agent was observed, which indicates the transition in its structure from three-dimensional framed silicon-oxygen skeletons to simpler one – chain, ribbon, layered ones which is evidenced with the interaction of PVP with chains of silicate particles .

At the same time, the use of polyvinylpyrrolidone as a modifier leads to slight decrease in the number of active centers on the surface of the nucleating agent and its specific area of the active surface relatively to acid-alkali indicator – methylene blue. This is probably owing to the blockage of active groups of the surface of silicate formations (silandiol, silanol, siloxane) through the physico-chemical interaction with the PVP functional groups [24].

Such changes in the surface characteristics of the nucleator in some way are confirmed by the results of IR spectroscopic studies (Fig. 1).
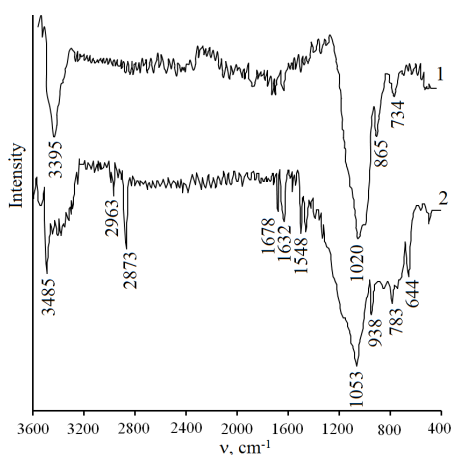

Fig. 1. FTIR spectra of Ni-containing nucleating agents: unmodified NA (1) and NA modified by PVP (2)

For the silicate Ni-containing nucleating agent the characteristic absorption bands with the highest intensities in the range of 1100–900 and 800–600 cm⁻¹ should be noted. These bands are caused by the internal main oscillations of the atoms in $[SiO_4]^-$ tetrahedrons, as well the valence asymmetric, symmetric, and deformation vibrations of the Si–O and O–Si–O bonds [25].

At the same time, the nucleating agent modified by polyvinylpyrrolidone is characterized by the presence of displaced typical PVP absorption bands: –CH_2– 2970–2860 cm⁻¹, C=O 1678 cm⁻¹, N–C=O 1632 cm⁻¹ [25]. The obtained data confirm the presence of polyvinylpyrrolidone in obtained materials, as well as its active participation in the process of modifying the surface of silicate nucleating agent.

For the modified nucleation agent, unlike the unmodified one, the less intense oscillation peaks of hydroxyl groups of hydration shells in the range of 3200–3600 cm⁻¹ are observed. The above mentioned is a consequence of the redistribution of intermolecular interactions in the system with the direct participation of the functional groups of PVP macromolecules and metal-silicon-oxygen frame. Such effect of the modifier promotes the increase of NA hydrophobicity and, as a consequence, the increase of its technological compatibility with a thermoplastic polymer matrix, polyamide in particular.

The peculiarities of the PVP effect on the Ni-NA structure are also confirmed by the obtained SEM photographs, which are shown in Fig. 2.

It was found that addition of the modifier – polyvinylpyrrolidone – during the synthesis of the nucleating agent provides the reduction in size of the individual particles and their agglomerates as well. Herewith, the particles obtained without PVP are characterized by their large size and solidity. Such features of the synthesized nucleating agents structure are explained by the PVP effect on the agglomeration stage of nanosized silicate particles during the interaction of sodium liquid glass and nickel chloride, in particular,

through inhibition of this process with its subsequent completion. The developed nucleating agents are distinguished by a number of characteristics which are required for polymer composites fillers: increased technological affinity for the polymer matrix; high purity degree, lack of impurities; required physical and chemical characteristics of the filler (size, dispersion, specific surface area, presence of functional groups, hydrophilic-

hydrophobic surface balance); stability of physical and chemical properties during the processing of composites under the influence of high pressure, temperature and aggressive environments. The developed nucleating agents were used as fillers-nucleators for polyamide 6.

The results of morphological studies based on X-ray structural analysis obtained for PA-6 composites filled with Ni-silicate nucleating agent are shown in Fig. 3.
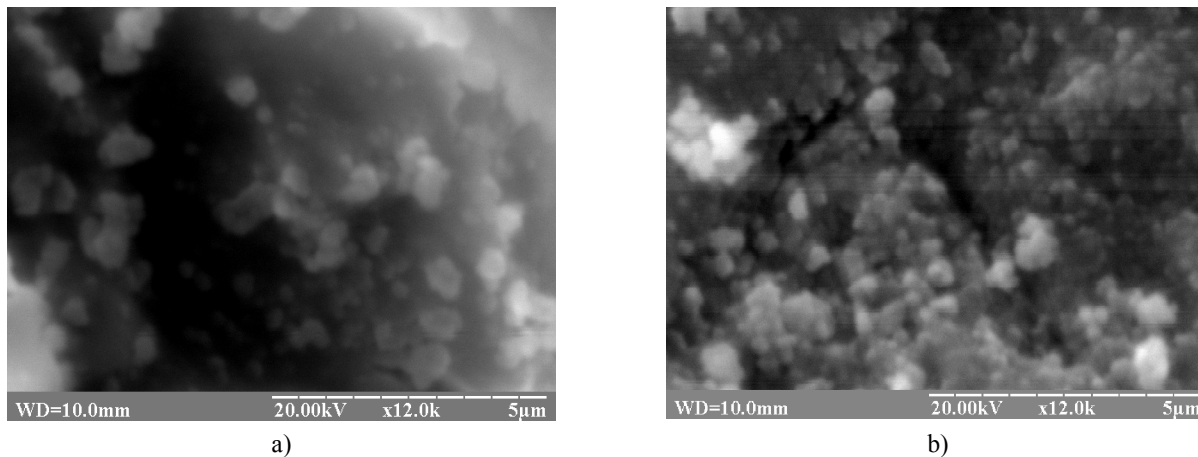

Fig. 2. SEM image of Ni-containing nucleating agent: unmodified NA (a) and NA modified by PVP (b)
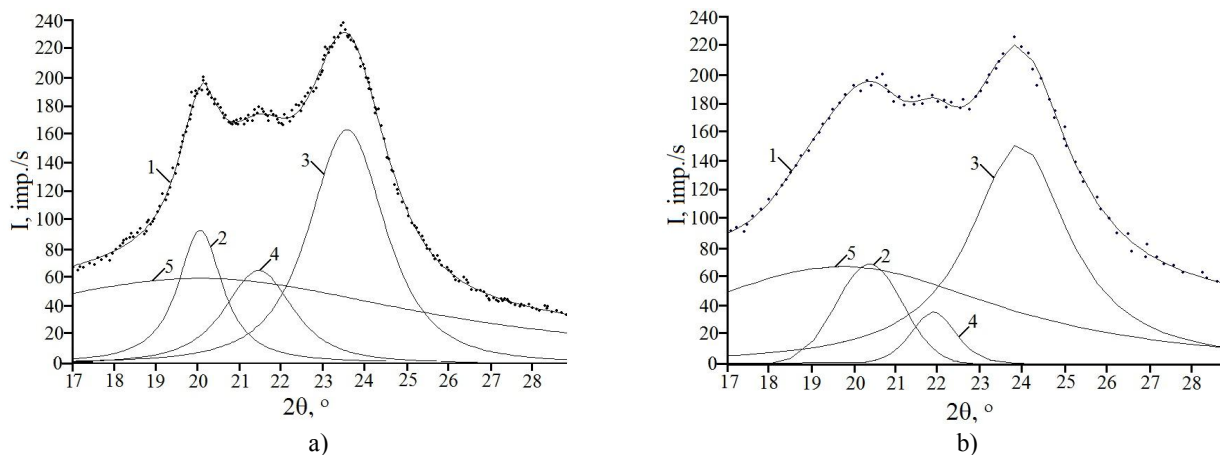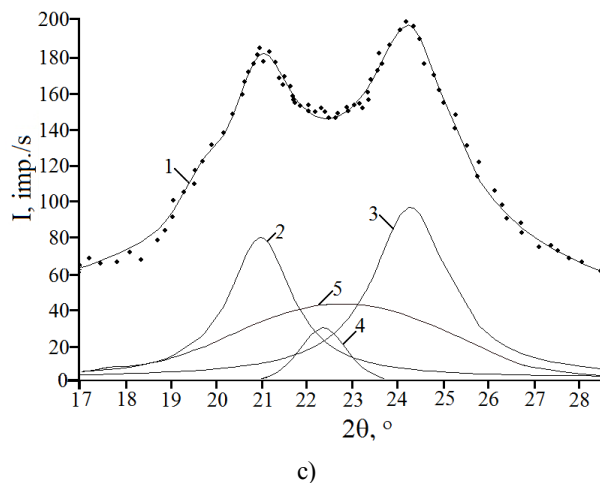

Fig. 3. Diffractograms of composites based on PA-6 without nucleating agent (a); with Ni-nucleating agent (b) and with Ni-PVP-nucleating agent (c). 1 is an approximation diffraction curve; 2, 3, 4, 5 are optimized curves of intensities of plane reflections (200) α-, (002) α-, γ- and amorphous phase

From the results of X-ray structural analysis of PA-6 materials (Fig. 3) it can be seen that the diffraction reflexes which are reflected from the crystalline phases of PA-6 are observed in the interval of the diffraction angle $2\theta = 18–26^o$. The general appearance of diffraction patterns of a composite based on PA-6 and Ni-containing nucleating agent and unfilled PA-6 is similar. In this case, the reflexes, which are reflected from the crystalline phases of pure PA-6, act at the diffraction angles $2\theta = 20.1$, 21.5 and 23.6^o, and the maximum of the amorphous phase is at the diffraction angle $2\theta = 20.0^o$.

The supramolecular structure of polyamide 6 is formed mainly by the antiparallel packing of the chains in the *ab* plane due to hydrogen bonds. Along the axis *c* of the chain they are packed in parallel and hydrogen bonds are absent. The diffraction reflexes filled PA-6 with the developed nucleating agent differ from the pure PA-6 by the configuration of the areas under the diffraction curves. In particular, for an unfilled PA-6, the reflex intensity of the plane *ab*- $(002)\alpha$ is greater than the reflex of the plane $(200)\alpha$ along the axis *c*. This is caused by the fact that during the melt cooling in the mold the fixing of hydrogen bonds between adjacent macromolecules occurs. Therefore, in the plane $(002)\alpha$ along the axis *ab* there is an antiparallel packing of the chains [26]. At the same time, additive of Ni-containing nucleating agent, especially modified by polyvinylpyrrolidone reduces the intensity of the reflexes of the planes *ab*- $(002)\alpha$, with the reflexes of the planes $(200)\alpha$ changed much less. This is, obviously, the consequence of redistribution of intermolecular interactions involving hydrogen bonds between the polyamide 6 and the functional surface groups of the nucleator and PVP during the PA-6 crystallization. In the composites with Ni-PVP-NA, the intensity of γ-phase reflexes is significantly reduced, which indicates a significant change in the crystallite size, and is obviously related to the large volume of PVP macromolecules having a helicoidal (spiral) conformation.

The structural parameters of the obtained materials: degree of crystallinity (Sc), crystallite size (L^1_{hkl}, L^2_{hkl}), interplanar distances (d_{hkl}) at the diffraction angle of crystalline peak (2θ), as well as peak height and width were determined using the WAXSFIT [18] software packages. They confirm the influence of the polymer-silicate filler on the supramolecular structure of PA-6. The data obtained are shown in Table 2.

For the composites based on PA-6 and modified Ni-containing NA the increase in crystallinity is significant, in particular, the crystallinity increases by 22.8 %. This is caused by the fact that the presence of polar silicate functional groups (siloxane, silanol and silandiol), carbamate groups of PVP macromolecules increases the possibility of antiparallel packing of the PA-6 chains and promotes increased interaction with the participation of hydrogen bonds between PA-6 amide groups and polyvinylpyrrolidone functional groups.

Based on the revealed influence of Ni-PVP nucleator on the morphological features of polyamide composites, it is expedient to define its operational properties. The working properties of composites based on thermoplastic polymers, in particular polyamide 6, significantly differ from unfilled thermoplastics. For polymer composites these properties can be broadly controlled by the physical condition and nature of the initial components, the nature of the distribution of ingredients in the volume of material, heat treatment, processing technological parameters and the like. In connection with this, researches of the main physico-mechanical and thermophysical properties of materials based on PA-6 and the developed nucleator are of great interest.

Table 2

Structural parameters of materials based on PA-6 and Ni-containing nucleating agent

Nucleating agent	S_c, %	Plane	2θ, ^{0}	Height peak	Width peak	L^1_{hkl}, Å	L^2_{hkl}, Å	d_{hkl}, Å
Without nucleating agent	47.0	$(200)\,\alpha$	20.1	92.8	1.3	70.7	49.7	4.4
		$(200)\,\gamma$	21.5	64.7	1.9	46.6	36.7	4.2
		$(002)\,\alpha$	23.6	163.3	2.1	42.2	33.0	3.8
Ni-NA	61.5	$(200)\,\alpha$	20.4	69.3	1.8	48.9	45.8	4.4
		$(200)\,\gamma$	21.9	36.2	1.2	72.4	62.8	4.1
		$(002)\,\alpha$	23.9	152.1	2.8	32.0	21.0	3.7
Ni-PVP-NA	69.8	$(200)\,\alpha$	20.3	95.4	1.1	64.5	52.8	4.4
		$(200)\,\gamma$	21.6	125.4	2.3	38.7	25.1	4.1
		$(002)\,\alpha$	23.9	64.1	1.6	20.0	13.9	3.7

The physico-mechanical properties of composites are one of the most important operational characteristics and largely determine the scope of the material and use conditions of the products based on them. Mechanical tests under static loads applying a voltage-elongation curve allow determining the basic tensile strength. It was determined that the stretching curves of polyamide materials are typical of crystalline polymers.

In particular, there are areas corresponding to a certain state of the supramolecular structure of the material under the action of the applied load. Based on the analysis of the tensile curves, the tensile strength (σ_{ts}) and the tensile elongation (ε_t) were determined. These characteristics, as well as the values of surface hardness before (F) and after (F_T) heat treatment for composites based on PA-6 are shown in Table 3.

The maximum value of tensile strength is observed for composites with modified polyvinylpyrrolidone NA which are characterized by the highest degree of crystallinity. At the same time, the increase in the strength of the developed materials also consists in the formation of additional bonds of PA-NA and PA-PVP which are destroyed under deformation and again restored in a new position. As a result, the equalization of local overstress takes place. An additional contribution to the enhancement is the increased mechanical hysteresis caused by the decrease in the mobility of macromolecules near the surface of Ni-containing PVP-NA and through the destruction of the PA-NA and PA-PVP bonds. Due to the increased hysteresis the degree of stress relaxation in polymer systems with a nucleator is greater than in the systems without it, especially in the area of large deformations at the top of growing cracks. The change in PA-6 morphology under the influence of the filler, in particular the increase in the degree of crystallinity and the decrease in the thickness of the interfacial layers, also confirm the values of surface hardness of the composites, which increase with application of modified nucleators and after heat treatment of the material. The increase of surface hardness after heat treatment also indicates the ordering of supramolecular structures.

Elastic, highly elastic and plastic properties are of great importance for polymer composite materials of structural and thermal applications, along with their strength characteristics. These properties were determined according to the module-deformation principle of calculation. Research of the deformational properties of PA-6-based composites is necessary to understand the behavior of a material under load of various types (static or dynamic, short-term or long-term, *etc*.) and to correctly select the field of application. The results of studies of elastic-deformation properties for composites based on PA-6 and Ni-containing polymer-silicate NA are shown in Table 4.

Table 3

Physico-mechanical properties of composites based on PA-6

Nucleating agent	σ_{ts}, MPa	ε_t, %	F, MPa	F_T, MPa
Without nucleating agent	58.4	220	328.5	357.4
Ni-NA	69.6	45	344.3	390.1
Ni-PVP-NA	79.9	40	397.6	415.8

Table 4

Elastic-deformational properties of composites based on PA-6

Parameter	Nucleating agent		
	–	Ni-NA	Ni-PVP-NA
Deformation module E_g, MPa	1033	1195	1227
Equilibrium modulus of elasticity E_{eq}, MPa	2718	3134	3469
Conditional instant modulus E_0, MPa	4059	4613	5207
Highly-elasticity module E_h, MPa	8228	9779	10397
Share of the elastic component in the total deformation ε_e, fraction	0.254	0.259	0.235
Share of highly elastic component in the total deformation ε_h, fraction	0.125	0.122	0.118
Share of the plastic component in the total deformation ε_p, fraction	0.619	0.618	0.646
Structure coefficient K_{str}	9.505	12.157	13.975

For composites with Ni-PVP-nucleating agent the highest value of the deformation modulus (E_g) is observed, which indicates a decrease in its ability to deform under conditions of static loads. The value of elasticity modulus which is characterized by the change of interatomic distances in the chains of macromolecules and deformation of the valence angles significantly increases during addition of NA, especially modified by polyvinylpyrrolidone. This is, obviously due to the decrease in the mobility of polyamide segments and macromolecules owing to interfacial interactions with the surface of the fine nucleator and active PVP groups. In addition, for polyamide composites there is a significant increase in the modulus of high elasticity, which is characterized by changes in the conformations of macromolecules and the restructuring of the spatial grid, which leads to a decrease in elasticity of the material. Such changes in the elastic-deformation characteristics of the developed composites indicate the enhancing effect of

NA, which leads to the limitation of the mobility of PA-6 macromolecules and the formation in the force field around the particles of thin adsorption layers of ordered supramolecular structures.

The structure coefficient of thermoplastic composites is a characteristic of a three-dimensional fluctuation network and characterizes to some extent the interphase and intermolecular interactions between the components. The NA addition leads to the increase in the coefficient of structure, in particular its greatest values are characteristic of composites with a modified nucleating agent, which is associated with its increased technological compatibility with PA-6 due to the intermolecular interactions of PA-6-PVP.

Important operational characteristics of polymer composites are their thermophysical properties. The influence of NA on Vicat softening point (T_V) and coefficient of linear thermal expansion of the developed materials (α_κ) were determined (Fig. 4).
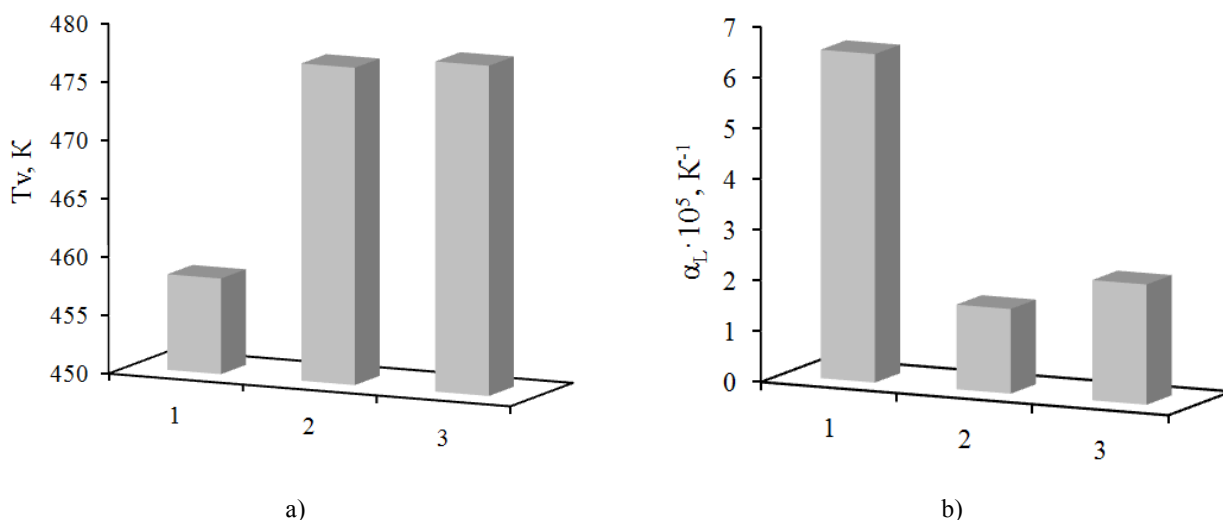

Fig. 4. Values of Vicat softening point (a) and coefficient of linear thermal expansion (b) of materials based on PA-6 without a nucleating agent (1); with Ni-nucleating agent and with Ni-PVP-nucleating agent (3)

As we can see, addition of a nucleating agent to PA-6 promotes the increase of Vicat softening point. For PA-6 and Ni-PVP-NA composites the highest T_V value is observed. This is apparently due to the increased compatibility of the NA modified by polyvinylpyrrolidone with PA-6 macromolecules as well compaction of the composite structure. One of the significant disadvantages of polymers and composites based on them is high coefficient of thermal expansion, which causes unacceptably large changes in the size of their parts, with significant fluctuations in temperature. Addition of Ni-nucleating agent promotes reduction of linear thermal expansion coefficient value of the developed materials (Fig. 4b). Such patterns are, obviously, conditioned by the fact that the crystalline

areas of the polymer are less exposed to thermal deformation compared to the amorphous areas and prevent the thermal expansion of the material. In this case, the lowest values of α_k are characteristic of composites with unmodified NA. Such features are primarily due to changes in the boundary layers at the division limit of the nucleating agent-thermoplastic matrix.

4. Conclusions

Developed Ni-containing polymer-silicate nucleators are highly efficient for polyamide materials. Based on X-ray diffraction analysis a significant influence

of Ni-containing nucleating agents on the supramolecular structure of polyamide 6 has been determined.

Due to uniform distribution of the modifier (polyvinylpyrrolidone) in it, as well as oriented influence on NA surface properties and increased technological compatibility between the components, polyamide 6 with Ni-containing polymer-silicate nucleator is characterized by increased crystallinity degree (by 15–22 %) and reduced average crystallite size. Materials based on polyamide 6 and modified nucleator possess increased physico-mechanical (surface hardness increases by 25–35 %, tensile strength by 30–35 %, modulus of elasticity by 25–35 %) and thermophysical (Vicat softening point increases by 15–20 K, coefficient of linear thermal expansion by 3–4 times) properties.

Improved physico-mechanical, elastic-deformational and thermophysical properties of the developed composites provide their effective application for manufacturing of structural and thermal engineering products in different industries working under conditions of high mechanical and thermal loads.

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ВПЛИВ ПОЛІМЕР-СИЛІКАТНОГО НУКЛЕАТОРА НА СТРУКТУРУ І ВЛАСТИВОСТІ ПОЛІАМІДУ 6

Анотація. *Наведені фізико-хімічні властивості розроблених нікель-вмісних модифікованих силікатних матеріалів як нуклеаторів для композитів на основі поліаміду 6. Виявлено, що введення нуклеатора модифікованого полівінілпіролідоном сприяє збільшенню ступеня кристалічності поліаміду 6 та зменшенню розмірів кристалітів. Встановлено, що зміни в структурі матеріалу сприяють збільшенню його міцності під час розривання, поверхневої твердості і теплостійкості за Віка.*

Ключові слова: *нуклеатор, полівінілпіролідон, кристалізація, поліамід 6, композит.*