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РОЛЬ МЕХАНОАКТИВАЦИИ НАНОЧАСТИЦ ТЕХУГЛЕРОДА В ФОРМИРОВАНИИ СЕГРЕГИРОВАННОЙ СТРУКТУРЫ И СВОЙСТВ ПОЛИМЕРНЫХ ЭЛЕКТРОПРОВОДЯЩИХ НАНОКОМПОЗИТОВ

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Исследованию влияния механоактивации наночастиц на структуру и свойства полимерных композитов в литературе уделено весьма недостаточное внимание. В ряде случаев это обстоятельство приводит к получению противоречивых сведений. Поэтому в данной работе рассмотрено влияние механоактивации наночастиц технического углерода марки Printex XE 2B на разрушающее напряжение, относительное удлинение и электропроводность нанокомпозитов на основе полиолефинов. В качестве полиолефинов использовали полиэтилен высокой плотности, полиэтилен низкой плотности, сополимер этилена с гексеном, сополимер этилена с бутеном, изотактический полипропилен, статистический рандом сополимер полипропилена, блок сополимера этилена с пропиленом. Показано, что в результате механоактивации наночастиц технического углерода в центробежной мельнице наблюдается снижение насыпной плотности практически в 12 раз. Приводится ряд предположений, объясняющих сам факт уменьшения насыпной плотности. Приведены данные по свойствам до и после механоактивации. Показано, что механоактивация по-разному влияет на характер изменения электропроводности. Установлено, что чем выше кристалличность исходной полимерной матрицы, тем больше проявляется эффект сегрегации и при более низкой концентрации технического углерода наблюдаются высокие значения цепочечной электропроводности в межфазной области. В то же время, механоактивация в присутствии компатибилизатора способствует некоторому увеличению разрушающего напряжения и относительного удлинения нанокомпозитов. Рассмотрено предположение о том, что в результате механоактивации наночастиц последние электризуются. Предполагается, что в процессе размола минеральных материалов происходит рост свежеобразованных поверхностей, которые обладают высоким энергетическим потенциалом, способным улучшить взаимодействие на границе раздела фаз полимер-наполнитель.

Ключевые слова: механоактивация, технический углерод, компатибилизатор, электропроводность, разрушающее напряжение, относительное удлинение

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THE ROLE OF MECHANOACTIVATION OF CARBON BLACK NANOPARTICLES IN THE FORMATION OF SEGREGATED STRUCTURE AND PROPERTIES OF POLYMERIC CONDUCTIVE NANOCOMPOSITES

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The study of the influence of mechanical activation of nanoparticles on the structure and properties of polymer composites has received very little attention in the literature. In some cases, this circumstance leads to the receipt of contradictory information. Therefore, in this paper, the effect of mechanical activation of Printex XE 2B carbon black nanoparticles on the tensile strength, elongation at break and electrical conductivity of polyolefin-based nanocomposites is considered. The polyolefins used were high-density polyethylene, low-density polyethylene, ethylene-hexene copolymer, ethylene-butene copolymer, isotactic polypropylene, statistical random polypropylene copolymer, and ethylene-propylene block copolymer. It has been shown that as a result of mechanical activation of carbon black nanoparticles in a centrifugal mill, a decrease in bulk density by almost 12 times is observed. A number of assumptions are given to explain the very fact of a decrease in bulk density. Data on properties before and after mechanical activation are provided. It has been shown that mechanical activation has different effects on the nature of changes in electrical conductivity. It has been established that the higher the crystallinity of the initial polymer matrix, the more pronounced the segregation effect is, and at a lower concentration of carbon black, high values of chain conductivity are observed in the interphase region. At the same time, mechanical activation in the presence of a compatibilizer contributes to some increase in the tensile strength and elongation at break of nanocomposites. The hypothesis that as a result of mechanical activation of nanoparticles, the latter become electrified is considered. It is assumed that during the grinding of mineral materials, the growth of newly formed surfaces occurs, which have a high energy potential, capable of improving the interaction at the polymer-filler phase boundary.

Keywords: mechanical activation, carbon black, compatibilizer, electrical conductivity, tensile strength, elongation at break

Polyolefins are finding increasing practical use in such industrial areas as mechanical engineering, aircraft manufacturing, automotive manufacturing, electronics, military and space technology. The reasons for this are the low density in relation to metals, chemical resistance, high physical and mechanical properties and the ability to be processed on high-performance equipment using methods of injection molding, extrusion, vacuum-pneumatic forming, etc. [1, 2]. In addition, polyolefins can significantly change their properties as a result of modifying their structure by introducing mineral fillers, stabilizers, cross-linking agents, polymers, etc. [3]. At the same time, considerable attention in the literature is given to studies on the selective improvement of any properties of polyolefins as a result of the introduction of dispersed and nanoparticles of various types of minerals and fillers [4, 5]. The use of nanotechnology has made it possible to significantly expand the range of changes in the properties of polyolefins and, thus, the areas of their application in various operating conditions [6-8]. In this regard, it should be noted that there is great interest in the problems associated with obtaining electrically conductive nanocomposites based on polyolefin dielectrics [9-11]. Research conducted in this direction has shown that the properties of polymeric materials are subject to change not only as a result of the introduction of various types of fillers and ingredients, but also as a result of the formation of a segregation (phase-separating) structure [12]. The term "segregation" is suitable for interpreting the structural features of semi-crystalline polymers, in particular polyolefins. At the same time, this term is not applicable to synthetic rubbers, which are known to be characterized by an amorphous structure. It is the segregation structure of semicrystalline polyolefins that is responsible for changing the properties of composites based on them [13-15]. It seems possible to obtain answers to many questions related to establishing the relationship in the “polymer matrix-filler-composite” system. As our studies in this area have shown, the properties of the composite material also depend on the preliminary mechanical activation of the filler particles [16-18]. As a result, questions arise which, based on existing ideas about the structure and properties of polymer composites, are difficult to fully scientifically interpret.

In this regard, the aim of this work is to study the influence of mechanical activation on the main physical and mechanical properties and electrical conductivity of nanocomposites based on polyolefins and carbon black.

EXPERIMENTAL PART

The following polyolefins were used as a polymer matrix:

A) **High density polyethylene (HDPE) – grade PETİLEN YY I668 (UV) (PETKIM) –** tensile strength 31.3 MPa, bending strength 34.4 MPa, elongation at break 435%, density 965 kg/m^3, Vicat softening temperature 139 °C, melting temperature 145 °C, crystallinity degree 82%, melt flow index 5.6 g/10 min.

B) **Low density polyethylene (LDPE) – 108-14 (Moscow) –** density 923 kg/m^3, melt flow index 7.8 g/10 min at load 5 kg, tensile strength 9.6 MPa, bending strength 16.2 MPa, elongation at break 360%, Vicat softening temperature 85 °C, melting temperature 101 °C, crystallinity degree 59%.

C) **Isotactic polypropylene (PP) – grade HP500M (SOCAR–POLYMER) –** tensile strength 33.0 MPa, elongation at break 30%, Vicat softening temperature 160 °C, melting temperature 169 °C, density 903 kg/m^3, crystallinity degree 65%, melt flow index 3.6 g/10 min.

D) **Random polypropylene copolymer (RPP) – grade RP2400 (OPF ATEX) –** thermoplastic random copolymer of ethylene and propylene with the following characteristics: tensile strength 28.5 MPa, elongation at break 600%, melt flow index 1.78 g/10 min, density 904 kg/m^3, melting temperature 146 °C, Vicat softening temperature 131 °C crystallinity degree – 57%.

E) **Block copolymer of ethylene with propylene (BEP) grade HB240R (BEP)** with the following properties: tensile strength – 25.6 MPa, elongation at break – 200%, bending strength 26.5 MPa, MFI – 2.9 g/10 min, Vicat softening temperature – 148 °C, melting temperature – 155 °C. BEP is a chain of propylene molecules interrupted by a chain of ethylene-propylene copolymer, the degree of crystallinity is 60%.

F) **Ethylene-hexene copolymer (EHC) – grade PE6438R (PAO Nizhnekamskneftekhim) –** linear low density polyethylene with the following characteristics: density 936 kg/m^3, tensile strength 37.4 MPa, elongation at break 810%, modulus of elasticity in bending 712 MPa, melting temperature 122 °C, Vicat softening temperature 115 °C, melt flow index 3.2 g/10 min, crystallinity degree 75%.

G) **Ethylene-butene copolymer (EBC) – grade PE4133Q** is characterized by the following properties: tensile strength – 27.1 MPa, elongation at break – 880%, density – 942 kg/m^3, modulus of elasticity in bending – 532 MPa, MFI – 4.6 g/10 min., melting temperature – 128 °C, Vicat softening temperature – 116 °C, crystallinity degree – 71%.

Compatibilizers:

- (PE-g-MA) - Maleic anhydride functionalized HDPE Exxelor PE1040 (ExxonMobil Chemical), designed to improve the compatibility of carbon black with all types of polyethylene.

- (PP-g-MA) - Exxelor PE1020 (Vanderbilt Chemicals, LLC), functionalized with maleic anhydride PP (PP-g-MA), is designed to improve the compatibility of carbon black with polypropylene and random copolymer polypropylene. The degree of grafting of maleic anhydride in both compatibilizers is 2.0-6.0 wt%.

The filler used was Printex XE 2B (Orion Engineered Carbons) carbon black (CB), a highly structured amorphous carbon black with particles measuring 18-20 nm. This filler is designed to improve the electrical conductivity of the polymer composite, as it is an electrically conductive CB, which is ideal for thermoplastic polymers, coatings, adhesives and sealants, natural and synthetic elastomers. The specific surface area is 800-1000 m^2/g density is 0.3-0.35 g/cm^3.

The particle size of carbon black and graphite was determined using a Mastersizer-3000 laser diffraction analyzer (Malvern). The method is based on measuring the angular dependence of the intensity of scattered light when a laser beam passes through a dispersed sample. Particle size determination range 0.01-3000 μm.

The tensile strength, yield strength and elongation at break of polymer composites were determined in accordance with GOST 11262-80. The stretching speed of samples on a tensile testing machine is 50 mm/min.

The electrical conductivity of the composites was determined in accordance with GOST 20214–74 "Electrically conductive plastics". The pressing of polymer samples for testing was carried out at a temperature of 160 °C for 4-6 min until a film with a thickness of 150 μm was obtained. Measurement of volume electrical resistance (for further calculation of specific volume resistance ρ and specific volume electrical conductivity σ) on direct current was carried out using an E6-13A teraohmmeter (OPF METROTEX). The thickness of the samples was measured using an IZV-2 optical measuring device. The experimental data were processed using Microsoft Excel 2016.

Mechanical activation of CB nanoparticles was carried out on a planetary mill of the XQM-2A brand from TENCAN. The device allows obtaining nanoparticles of mineral fillers from 10 to 100 nm. The maximum grinding time is 8 minutes. With a longer grinding time, CB changes in particle size are practically not observed.

Preparation of nanocomposites. In order to modify the properties of polyolefins, up to 2.0 wt % of the compatibilizer PE-g-MA or PP-g-MA was first introduced into their composition on hot rollers at a temperature of 150-170 °C, and then CB was added in parts to the molten polymer mixture. The rolling time was 10 min. The friction of the rollers was equal to 1.29.

To achieve the greatest dispersion of nanoparticles in the volume of the polymer matrix of the nanocomposite, the mixture obtained on the rollers was remixed on a laboratory twin-screw extruder with counter-rotation of Chinese manufacture, brand SJZS-10A. Mixing of the mixture components in the melt mode was carried out in the temperature range of 150-210 °C, while the number of screw revolutions was 60 rpm. The objective of the study was to conduct a preliminary assessment of the dispersion of nanocomposites under conditions of intensive mixing in the melt mode.

RESULTS AND DISCUSSION

Dispersion and mechanical activation have a great influence on the surface properties of minerals and rocks [19, 20]: a noticeable change in the physical properties and chemical activity of the substance occurs. This is explained not only by an increase in the specific surface area and a decrease in particle size, but also by a change in structure, in particular, the amorphization of surface areas due to the occurrence of mechanochemical processes. The study of this issue in the literature is given very little space, despite the fact that mechanical activation of nanoparticles can significantly affect not only their dispersion and bulk density, but also the electrical conductivity and properties of nanocomposites. A number of points were identified that are not always possible to interpret by referring only to existing scientific provisions. For example, as a result of mechanical activation of CB nanoparticles for 8 min, an increase in their volume was established from almost 20 to 240 ml, i.e. 12 times (Figure). At the same time, the bulk density decreased from 325 kg/m^3 to 27 kg/m^3.

Fig. The influence of mechanical activation of CB nanopowder on its volume and bulk density

Рис. Влияние механоактивации нанопорошка ТУ на его объем и насыпную плотность

It was found that such a sharp increase in the volume of CB nanopowder after mechanical activation can be interpreted as further grinding of nanoparticles as a result of the destruction of aggregates into smaller particles. It was experimentally established that when the grinding time is over 8 min, the particle size and volume of the powder remain virtually unchanged. In addition, the volume of mechanically activated powder in the measuring cup (Figure) did not undergo any changes during the year. There is reason to believe that during the milling of CB nanoparticles, new and resistant to change freshly formed surfaces (chips) are formed, which have a high energy potential capable of improving interaction at the phase boundary [21, 22]. In addition, when processing CB nanoparticles, the cause of mechanical activation is a complex of processes, of which, apparently, one can single out the accumulation of structural defects by nanoparticles, collision and friction of particles, as well as the appearance of active centers on the freshly formed surface during the destruction of solid particles. These processes include the catalytic effect of electrons, the flow of which occurs at the mouth of the crack at the moment of particle grinding [19].

To improve the structuring properties, CB nanoparticles were subjected to mechanical activation in a planetary mill. And, indeed, when we assessed the size of mechanically activated CB nanoparticles, we found a slight decrease in their size from 25 nm to 15-20 nm. However, the results of the study of the effect of mechanical activation on electrical conductivity were ambiguous.

The decrease in bulk density of CB after mechanical activation can be explained by three main reasons:

1 - during long-term storage, powder nanoparticles become stale and stick together to form large agglomerates of particles, which, under mechanical action, are crushed and dispersed into smaller particles - aggregates, which are covalently bonded CB nanoparticles;

2 - during the process of mechanical activation, the size and shape of the grains of the mixture become approximately the same, which leads to an increase in intergranular voids per unit volume. Previously, the authors [20] established the possibility of using bulk density as an additional criterion when assessing the fineness of grinding of binders;

3 - we do not exclude the possibility of electrification of nanoparticles under the influence of mechanical activation and destruction of the structure of covalently bound CB nanoparticles in aggregates. Electrification of nanoparticles can promote an increase in surface energy and their mutual repulsion, resulting in an increase in the volume of the powder and, as a consequence, a decrease in its bulk density.

Table presents the results of the study of the effect of mechanical activation on electrical conductivity and some physical and mechanical properties of compatibilized nanocomposites. The compatibilizer was used exclusively to improve the compatibility of the polymer matrix with CB nanoparticles. From a comparative analysis of the data in this table, it can be established that as a result of mechanical activation, some increase in the electrical conductivity of CB-filled nanocomposites is observed. A noticeable increase was also observed in the assessment of the strength characteristics and elongation at break of nanocomposites. As can be seen from the table, with a relatively high filler content in the nanocomposite, the mechanical activation effect is somewhat reduced. It should be noted that the most noticeable effect of mechanical activation on the electrical conductivity of nanocomposites is manifested at low concentrations of CB (5.0-10 wt. %).

When introducing 15–20 wt. % CB into the composition of nanocomposites, mechanical activation has an ambiguous effect on the change in electrical conductivity. For example, those samples of the original polyolefin that are characterized by relatively high crystallinity degree (HDPE, EHC and EBC), with a filler concentration of over 10 wt. %, are practically not susceptible to the effect of mechanical activation on electrical conductivity. This circumstance is due to the fact that at relatively high concentrations of CB, the interphase region is saturated with nanoparticles, resulting in chain electron conductivity. Therefore, a further increase in the content of mechanically activated nanoparticles has virtually no effect on the value of this indicator [23]. When using polyolefins with a relatively low degree of crystallinity (LDPE*, PP*, RPP*) as a polymer matrix, the process of formation of chain electron conductivity in the interphase region constantly increases with an increase in the filler content [23]. This explains that in such polymer matrices the efficiency of mechanical activation constantly increases with increasing CB content. Apparently, in this case it will be appropriate to emphasize that the effect of mechanical activation in polyolefins is largely determined by the structure and degree of crystallinity of the original polymer matrix. We have shown above that the degree of crystallinity of HDPE is 82%, LDPE – 59%, PP – 65%, EHC – 75%, EBC – 71%, RPP – 57%, BEP – 60%. For example, if the original polymer matrix is HDPE, then only 18% (approximately 1/5 of the total volume) is accounted for by the amorphous region. It was noted above that the filler particles in the HDPE composition are distributed predominantly in the interspherulitic amorphous region. Therefore, the introduction of even 5.0 wt. % CB into the composition of HDPE will lead to the fact that in the interspherulitic amorphous region its concentration will increase approximately 5 times, i.e. will be 25 wt. %, at which electronic conductivity will be achieved. In the case of using LDPE as a polymer matrix, 41%, i.e. approximately 2/5, is accounted for by the amorphous region. This means that the introduction of 5.0 wt. % CB into the composition of LDPE will lead to an increase in the filler concentration in the interspherulitic region to only 12.5 wt. %. As shown in Table 1, at such a concentration of TU in the composition of LDPE, the maximum chain electron conductivity is not ensured. According to the data in Table 1, such electron conductivity in LDPE is achieved with the introduction of 10 wt. % CB, i.e. when the filler content in the interspherulite region reaches the threshold value of 25 wt. %. Therefore, the lower the degree of crystallinity of the polyolefin, the more CB must be introduced to achieve the maximum effect in improving the electrical conductivity of nanocomposites [8, 23, 24]. This principle is also maintained when introducing mechanically activated CB nanoparticles.

Table

Properties of conductive nanocomposites based on compatibilized polyolefins and CB, before and after mechanical activation

***Таблица*. Свойства электропроводящих нанокомпозитов на основе компатибилизированных полиолефинов и ТУ, до и после механоактивации**

№	Composition of the nanocomposite, wt %	Electrical conductivity, (Om.m)$^{-1}$		Tensile strength, MPa		Elongation at break, %	
		before	after	before	after	before	after
1	HDPE*+5.0 CB	$1.2\cdot10^{-2}$	$5.6\cdot10^{-2}$	32.8	34.6	105	120
2	HDPE*+10 CB	$4.8\cdot10^{-2}$	$5.4\cdot10^{-2}$	32.6	35.2	55	85
3	HDPE*+15 CB	$6.5\cdot10^{-2}$	$6.9\cdot10^{-2}$	30.1	30.8	50	65
4	HDPE*+20 CB	$7.0\cdot10^{-2}$	$7.3\cdot10^{-2}$	28.5	29.1	35	35
5	LDPE*+5.0 CB	$4.6\cdot10^{-5}$	$3.2\cdot10^{-4}$	17.2	18.8	165	185
6	LDPE*+10 CB	$3.7\cdot10^{-2}$	$7.8\cdot10^{-2}$	17.5	19.4	120	135
7	LDPE*+15CB	$1.7\cdot10^{-2}$	$3.2\cdot10^{-2}$	17.8	18.5	95	110
8	LDPE*+20 CB	$3.3\cdot10^{-2}$	$4.1\cdot10^{-2}$	16.8	17.0	55	55
9	PP*+5.0 CB	$2.4\cdot10^{-7}$	$2.1\cdot10^{-6}$	35.6	36.8	100	120
10	PP*+10 CB	$3.7\cdot10^{-5}$	$9.3\cdot10^{-5}$	32.8	33.7	75	85
11	PP*+15 CB	$2.6\cdot10^{-4}$	$7.1\cdot10^{-4}$	31.2	31.6	75	75
12	PP*+20 CB	$3.1\cdot10^{-2}$	$3.9\cdot10^{-2}$	28.4	29.7	45	50
13	RPP*+5.0 CB	$2.3\cdot10^{-7}$	$8.6\cdot10^{-7}$	27.1	28.2	200	200
14	RPP*+10 CB	$7.4\cdot10^{-4}$	$0.9\cdot10^{-3}$	24.6	25.5	55	65
15	RPP*+15 CB	$2.5\cdot10^{-2}$	$3.0\cdot10^{-2}$	24.1	24.4	40	50
16	RPP*+20 CB	$3.0\cdot10^{-2}$	$3.8\cdot10^{-2}$	21.7	21.9	35	35
17	BEP*+5.0 CB	$6.1\cdot10^{-7}$	$9.2\cdot10^{-7}$	27.7	28.5	140	150
18	BEP*+10 CB	$8.3\cdot10^{-4}$	$0.9\cdot10^{-3}$	32.6	33.1	65	85
19	BEP*+15 CB	$3.3\cdot10^{-2}$	$3.9\cdot10^{-2}$	30.1	30.4	50	60
20	BEP*+20 CB	$4.3\cdot10^{-2}$	$5.0\cdot10^{-2}$	28.5	28.8	35	35
21	EHC*+5.0 CB	$7.3\cdot10^{-3}$	$1.9\cdot10^{-2}$	38.4	39.6	100	110
22	EHC*+10 CB	$4.3\cdot10^{-2}$	$5.9\cdot10^{-2}$	34.6	35.3	65	75
23	EHC*+15 CB	$6.5\cdot10^{-2}$	$7.0\cdot10^{-2}$	30.5	30.8	50	60
24	EHC*+20 CB	$6.9\cdot10^{-2}$	$7.1\cdot10^{-2}$	28.5	28.8	25	25
25	EBC*+5.0 CB	$7.3\cdot10^{-3}$	$9.1\cdot10^{-3}$	35.4	36.7	80	95
26	EBC*+10 CB	$5.7\cdot10^{-2}$	$6.9\cdot10^{-2}$	32.6	33.8	55	65
27	EBC*+15 CB	$6.3\cdot10^{-2}$	$6.6\cdot10^{-2}$	30.3	30.7	50	50
28	EBC*+20 CB	$7.5\cdot10^{-2}$	$7.7\cdot10^{-2}$	28.5	28.8	35	35

Note: *indicates compatibilized polymer

Примечание: *обозначает совместимый полимер

Mechanical activation has an ambiguous effect on the tensile strength and elongation at break of nanocomposites. Analyzing the data presented in Table 1, it can be established that the process of mechanical activation contributes to the increase of the tensile strength and elongation at break of nanocomposites. And in this case, the concentration effect is manifested in the mechanism of action of mechanical activation in the interphase amorphous region. As in the assessment of electrical conductivity, the effect of mechanical activation is more pronounced at filler concentrations within 5.0-15 wt. % in the composition of HDPE*, EHC*, EBC*. As for polyolefins with a comparatively lower degree of crystallinity (LDPE*, PP*, RPP*, BEP*), in these samples the effect of mechanical activation is also evident at higher CB contents.

Such a noticeable effect of mechanically activated nanoparticles on the increase of electrical conductivity and strength properties of nanocomposites is undoubtedly associated with the above three simultaneously possible reasons contributing to a 12-fold decrease in bulk density. This effect becomes possible due to the orientation of macrochains on the developed specific surface of activated nanoparticles, as a result of which the greatest adhesive contact with the polymer matrix is achieved with subsequent improvement of properties.

Of course, the analysis of literature on the problem of mechanical activation of mineral particles and their influence on the properties of nanocomposites is still in the early stages of research and, therefore, any attempts by scientists aimed at obtaining new scientific results will ultimately allow generalizing the research results into a single, integral theory.

CONCLUSIONS

The influence of mechanical activation on the properties of nanocomposites based on polyolefins and carbon black has been established. It is shown that as a result of mechanical activation, some increase in electrical conductivity, tensile strength and elongation at break of nanocomposites is observed.

Авторы заявляют об отсутствии конфликта интересов, требующего раскрытия в данной статье.

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