

UDC 678.01

TECHNOLOGICAL FEATURES OF INJECTION MOULDING OF DYNAMICALLY VULCANIZED NANOCOMPOSITES BASED ON RANDOM POLYPROPYLENE, NITRIL BUTADIENE RUBBER AND BENTONITE**A.J. Guliyev**

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*Received 10.06.2020**Accepted 16.09.2020*

Abstract: *The work deals with the influence of the technological parameters of injection molding on the basic physicomechanical characteristics of nanocomposites of dynamically vulcanized thermoplastic elastomers based on random polypropylene and nitrile butadiene rubber of various grades. The equipment used was a single-screw molding unit with 4 temperature zones of the material cylinder. Rotational and translational movement of the screw allowed for uniform dispersion of the mixed components of the mixture. To improve the compatibility of the polymer components, a graft copolymer of polypropylene with maleic anhydride at the value of 1-3 wt. % was used as compatibilizer. The influence of the temperature regime of the material cylinder of the injection machine and the molding pressure on the ultimate tensile stress, elongation at break, elastic modulus on bending and volume shrinkage is studied. It is shown that an increase in the indicated technological parameters of molding leads to an increase in strength indicators and a noticeable decrease in the volume shrinkage of dynamically vulcanized thermoplastic elastomer nanocomposites. The influence of mold temperature and holding time under pressure on the ultimate tensile stress and elongation at break of nanocomposites has been considered and optimal conditions for their cooling in the mold established. Besides, the effect of nanocomposites of dynamically vulcanized thermoplastic elastomers with various types of nitrile butadiene rubber on the ultimate tensile stress and elongation at break of nanocomposites analyzed. It revealed that, unlike random polypropylene, dynamically vulcanized nanocomposites are characterized by isotropic properties which make it possible to obtain high-quality structural materials of complex configuration.*

Keywords: *thermoplastic elastomer, nitril butadiene rubber, random polypropylene, bentonite, injection moulding*

DOI: *10.32737/2221-8688-2020-3-388-395*

Introduction

As the world industrial production of polymers is expanding, studies into the development of polymer composite materials capable of operating in harshly extreme conditions have intensified significantly. The loading of various mineral fillers, plasticizers, polymers with chemical modifications as a whole made it possible to obtain a whole set of polymer composites with high quality characteristics [1-5]. The most promising is the development of thermoplastic elastomers and dynamically vulcanized nanocomposites based on incompatible polymer mixtures. An interest in the use of bipolar components-based polymer mixtures is due to the fact that in the final product it is possible to reproduce the properties

of completely different in nature polymers. In this regard, polymer mixtures based on thermoplastic polyolefins and nitrile butadiene rubber are most interesting. The use of the method of dynamic vulcanization of polymer mixtures in the presence of nanoscale mineral fillers fundamentally changed the very idea of structure and properties of thermoplastic elastomers (TEP) [6,7]. Despite rise in the number of works on the subject, studies into technology of processing nanocomposites in dynamically vulcanized thermoplastic elastomers (DVEP), in particular by injection molding, are inappreciable. In connection with the foregoing, the aim of the work is to examine technological features of the processing of

dynamically vulcanized nanocomposites based on incompatible polymer mixtures.

Experimental part

Random polypropylene (RPP) or thermoplastic random copolymer of ethylene with propylene extrusion grade RP2400 is characterized by the following properties: ultimate tensile stress – 28,5 MPa, elongation at break – 600%, MFI = 1,78 g/10min., density 0,904g/cm³, melt point 146°C, heat resistance 131°C.

SKN-40 nitrile butadiene rubber with 40 wt. % acrylate nitrile content.

Exxelor PO1200 compatibilizer is meant to be used as a binding agent for improvement of compatibility of polymeric materials and intensification of reactivity when interacting with inorganic fillers. This is achieved by reactive extrusion by graft copolymerization of polypropylene with maleic anhydride (PPMA). The concentration of MA in the compatibilizer is 3.0 wt. %.

Bentonite (Bt) - is a mixture of natural aluminosilicate minerals, the basis of which is montmorillonite $Al_2O_3 \cdot 4SiO_2 \cdot n \cdot H_2O$ (60-70%). In bentonites, kaolinite and other minerals are present.

Bentonite nanoparticles with a size of 70-90 nm were obtained in an A-11 analytical mill at a maximum rotor speed of 30000 rpm.

The size of the nanoparticles was determined on a STA PT1600 Linseiz Germany model instrument.

Ultimate tensile stress and elongation at break of nanocomposites were determined by

the results of analytical data (5 measurements) in accordance with GOST 11262-80. The relative error of the experiment is 3-5%.

Bending modulus was determined in accordance with GOST 9550-81.

MFI of polymeric materials were determined on a MELT FLOW TESTER, CEAST MF50 capillary rheometer (INSTRON, Italy) at a temperature of 190 °C and a load of 5 kg. The relative error of the experiment is 5%.

To conduct studies for evaluation of physicomechanical properties of polymer compositions, samples were cast on a brand ДЕ3132.250IQ1 injection molding machine. The material cylinder of this equipment consists of 4 heated zones, a worm type screw (L / D ratio = 24) with preliminary plasticization which rotates and can simultaneously move forward and backward. This design of the injection machine allows you to use its capabilities at the same time for mixing the components of the mixture and casting the product in the mold.

Volumetric shrinkage was determined by measuring the change in the length of the bar from the original length of the molding part of the mold 55.5 mm. Shrinkage (Δ) was calculated by the equation: $\Delta = (55.5 - L) 100\% / 55.5$ where: L- is the current value of the sample length. The shrinkage value was determined as the average of 5 experiments conducted for each sample.

Results and discussion

Before starting on experimental research to optimize the technological aspects of the DVEP nanocomposites processing, it seemed interesting to focus on technological compatibility of polymer components. Governed by the principle that there are no thermodynamically compatible polymer systems, we built all studies on technologically compatible composites. Technological compatibility of polymers shows the ability of polymers to get mixed without delamination. To achieve this state, the common methods are those that use compatibilizers which at the interface provide high adhesion to

heterogeneous macrochains [8]. In this work, such heterogeneous components of the mixture are RPP and SKN-40. RPP is among nonpolar polymers, and SKN-40 is highly polar. In this regard, the selection of the compatibilizer was based on the use of grafted copolymers where the main chain is polypropylene (PP), and the grafted part is single units of maleic anhydride (MA). This circumstance is explained as being due to the fact that MA has no ability to polymerize and, therefore, is grafted with individual links to the PP macrochain. To our thinking, in the process of mixing the components of the mixture, the PPMA will be

distributed in the interfacial region, thereby contributing to rise in adhesive forces in the contact zone of the dispersed phase SKN macrochains with RPP macrochains. It was experimentally established that the amount of PPMA required to achieve the maximum effect from its use was 2.5-3.0 wt. %.

Thus, the injection molding process consists of three main stages: preliminary preparation of the feedstock, mechanochemical synthesis of DVEP nanocomposites and casting of high-quality products in the mold [9]. To accomplish this task, there are two options for technological solutions. According to the first variant, the prepared granulated DVEP nanocomposite obtained on the basis of RPP + 40 wt. % SKN-40 + 3.0 wt. % PPMA + 5.0 wt. % bentonite nanoparticles cross-linked 0.5 wt. % DP obtained as a result of reaction extrusion is used as a starting material. These granules are loaded into the hopper of an injection machine to obtain structural products based on them. The second option is used in the absence of granules. In this embodiment, all the initial components of the mixture are loaded simultaneously into the hopper of the injection machine. However, this approach is faced with certain difficulties arising from uneven dispersion of mixture components, including the DP crosslinking agent. At this option, bentonite, DP and PPMA will be pre-mixed on cold rollers with SKN-40. The resulting mixture SKN-40, after passing through a grinding unit, is crushed to a crumb, and then, after dry mixing with RPP, it is loaded into the hopper of an injection machine. Therefore, in this work, we will use materials obtained by both options as feedstock.

The design of the injection machine ДЕ3132.250И1 has the characteristic that it provides intensive mixing of the mixture components in question according to a single

technological scheme according to which it seems possible to obtain filled nanocomposites with uniform dispersion of solid nanoparticles in a polymer matrix. Along with this, it seems possible to carry out mechanochemical synthesis in it with the formation of DVEP nanocomposites [10]. Through changing the temperature of the material cylinder and the mold, the injection pressure makes it possible to influence, to a certain extent, changes in the physicochemical and technological properties of nanocomposites. The use of injection equipment reveals that after heating of polymer mass, the mixing process is accompanied not only by the rotation of the screw but by partial translational movement of the screw as well. In this case, it seems possible to create the necessary injection pressure into the mold. After the material is injected into the mold and the shutter speed is maintained under pressure, the screw moves back to its original position during constant rotation. Such a technological feature of the operation of the injection unit allows not only to reduce the residence time of the polymer in the material cylinder, but also significantly affect the increase in its productivity [10, 11]. Table 1 shows results of experimental studies into influence of the technological mode of injection molding on the basic physicochemical properties of DVEP nanocomposites obtained in line with the first embodiment. As noted above, according to this embodiment, the starting material is finished granules of DVEP nanocomposites. Analyzing the data presented in this table, it can be established that with an increase in the temperature regime of the material cylinder and casting pressure, a general tendency is observed to increase the ultimate tensile stress and the bending elastic modulus.

Table 1. Effect of temperature conditions and casting pressure on the properties of DVEP nanocomposites based on granular RPP + 40 wt. % SKN-40 + 3.0 wt. % PPMA + 5.0 wt. % bentonite

Temperature in zones, °C	Casting pressure, MPa	Ultimate tensile stress, MPa	Elongation at break, %	Shrinkage, %	Elastic modulus, MPa
130-150-160-170	50	23.8	40	0.25	1121
130-150-165-175		23.8	40	0.25	1125
140-150-170-180		24.0	50	0.25	1156
140-160-170-190		24.0	55	0.24	1160

130-150-160-170	100	24.0	50	0.18	1254
130-150-165-175		24.0	50	0.18	1272
140-150-170-180		24.7	50	0.15	1285
140-160-170-190		25.0	50	0.15	1290
130-150-160-170	150	25.0	50	0.16	1269
130-150-165-175		25.3	50	0.15	1278
140-150-170-180		25.3	50	0.15	1305
140-160-170-190		25.3	50	0.15	1316

The revealed regularity of growth in strength indicators can be interpreted as follows: as casting pressure and temperature rises, a significant compaction of the melt occurs in the molding part of the mold. The latter circumstance also contributes to noticeable decrease in volumetric shrinkage which is a very important technological indicator of DVEP for the production of structural products of complex configuration. The value of the elongation at break of DVEP nanocomposites is not significantly dependent on technological parameters of processing and, therefore, its value varies within 40-50%.

When comparing the properties of injection samples of DVEP nanocomposites with its initial values, i.e. before processing by injection molding, some changes in properties can be detected. For example, the initial granular DVEC nanocomposite has the following properties: ultimate tensile stress of 27.0 MPa, elongation at break of 60%. A certain decrease in the ultimate tensile stress from 27.0 to 25.3 MPa may be due to the fact that during the injection molding process a partial destruction of the polymer matrix occurs. However, these are just minor changes which suggest the possibility of using granular DVEP

nanocomposites in the process of injection molding.

Another important problem in injection molding is the study into the influence of mold temperature and holding time under pressure on basic physicomaterial properties of DVEP nanocomposites. At the same time it was interesting to consider the influence of the location of blade samples in relation to the mold gate. The need for such studies is due to the fact that the polymer material under consideration is poorly studied, especially in the field of their processing on standard equipment. In addition, any information related to the selection of optimal conditions for cooling the product in the mold is pressing and required in real production process.

Table 2 indicates results of experimental studies into the effect of mold temperature and holding time under pressure on the properties of DVEP nanocomposites. Analyzing the Table data, one can notice that the temperature-time regime of the mold makes certain adjustments in the properties of DVEP nanocomposites. In particular, with an increase in mold temperature from 25 to 75, there is a general tendency towards a slight increase in the ultimate tensile stress.

Table 2. Influence of mold temperature and holding time under pressure on the strength properties of nanocomposites based on granular DVEP: RPP + 40 wt. % SKN-40 + 3.0 wt. % PPMA + 5.0 wt. % bentonite. Casting pressure is 150 MPa. Casting temperature by zones is 140-150-170-180.

Mold temperature T, °C	Pressure holding time, sec	Ultimate tensile stress, MPa	Elongation at break, %
25	5.0	23.2	40
50		23.8	40
75		23.8	45
25	10	24.0	40
50		24.5	40
75		24.4	50

25	20	24.4	50
50		24.9	50
75		24.6	50
25	30	24.4	50
50		24.7	50
75		24.7	50

The temperature increase mainly helps to reduce the temperature difference "melt-mold" and thus decrease the cooling rate of the product. In some cases, this practice reduces internal tension in the product, preventing the possibility of warpage [12, 13]. A relatively low temperature of the mold, on the contrary, promotes rapid cooling, which does not allow sufficient relaxation of the macromolecular structure, which affects the accumulation and maintenance of internal tension in the product. At the same time, it was found that the most optimal is a 20 second exposure time under pressure. A further increase in the exposure time no longer leads to

any changes in the properties of DVEP nanocomposites.

Table 3 presents the experimental data on the influence of the blade samples location in the mold on the ultimate tensile stress and the elongation at break of DVEP nanocomposites. Analyzing the data presented in this Table, it can be established that the location of the samples in the mold (along and across) with ratio to the sprue leads to no significant changes in the ultimate tensile stress and elongation at break for samples based on DVEP nanocomposites.

Table 3. Influence of the blade location in the mold on physicomechanical properties of various types of DVEP nanocomposites under optimal temperature casting conditions (140-160-170-190 ° C), mold temperature 50 ° C and holding time 20 seconds.

Composition of the DVEP nanocomposite, wt. %	Location of the blades in the mold	Ultimate tensile stress, MPa	Elongation at break, %
RPP	along	28.5	600
	across	26.2	585
RPP+1PPMA+40SKN-18+5B	along	23.4	60
	across	23.3	60
RPP+3PPMA+40SKN-26+5B	along	24.5	50
	across	24.5	50
RPP+3PPMA+40SKN-40+5B	along	25.3	35
	across	25.3	40

B- bentonite nanoparticles

For the initial RPP, it is clearly seen that the samples located along the direction of the melt flow are characterized by relatively high values of the ultimate tensile stress. This is primarily due to orientation processes occurring in the polymer matrix melt along the flow direction. When passing to DVEP nanocomposites, the difference in the strength of the samples located along and across the mold gate almost completely disappears. This circumstance, to a certain extent, contradicts the generally accepted notions of orientation processes that affect the supramolecular structure and

properties of products in the mold [14,15]. However, if we proceed from the fact that the polymer material under consideration has a spatially cross-linked structure with gel fraction content of 25-35 wt. % to a certain extent, this circumstance allows us to give a different interpretation of the experimental data. In any case, it is obvious that polymeric materials having the rubber properties are characterized by highly elastic deformation with corresponding reversible deformation. Therefore, the rules that are established for thermoplastic polymers, such as RPP, cannot be

fully applied to materials with rubber properties. Considering that DVEP nanocomposites have rubber-like properties, we can state that DVEP nanocomposites have isotropic properties. A similar feature of the developed material is extremely important for obtaining structural materials of complex configuration. Thus,

based on the foregoing, it can be argued that the developed DVEP nanocomposites based on RPP and SKN can be processed by injection molding to obtain structural materials that have the properties of rubbers and can be processed as thermoplastics.

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RANDOM POLİPROPİLEN, BUTADİEN-NİTRİL KAUCUKU VƏ BENTONİT ƏSASINDA DİNAMİK VULKANLAŞMIŞ NANOKOMPOZİTLƏRİN TƏZYİQ ALTINDA TÖKMƏSİNİN TEXNOLOJİ XÜSUSİYYƏTLƏRİ

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Məqalədə təzyiqlə təzyiqlə altındakı tökmənin texnoloji parametrlərinin random polipropilen və müxtəlif markalı butadien-nitril kauçuku əsasında olan dinamik vulkanlaşmış termoelastoplast nanokompozitlərin əsas fiziki-mexaniki xüsusiyyətlərinə təsiri nəzərdən keçirilmişdir. Avadanlıq olaraq 4 temperatur zonalı material silindri birşəkli tökmə cihazından istifadə olunmuşdur. Şnekin fırlanma və qəbul hərəkəti qarışığın qarışdırılan komponentlərinin bərabər dispersləşməsinin həyata keçirilməsinə icazə vermişdir. Polimer komponentlərin uyğunluğunun artırılması üçün kompatibilizator olaraq polipropilenin 1-3 kütlə % miqdarında malein anhidridi calaq olunmuş bircə polimerindən istifadə olunmuşdur. Tökmə maşınının material silindrinin temperatur rejiminin və tökmə təzyiqinin dağıdıcı gərginlik, nisbi uzanma, əyilmədə elastiklik modulu və həcmi sıxılmaya təsiri öyrənilmişdir. Tökmənin qeyd olunan texnoloji parametrlərinin dinamik vulkanlaşmış termoelastoplast nanokompozitlərin möhkəmlik göstəricilərinin artımına, həcmi sıxılmasının nəzərəcarpacaq azalmasına səbəb olduğu göstərilmişdir. Pressformanın temperaturunun və təzyiqlə altındakı saxlanılma müddətinin nanokompozitlərin dağıdıcı gərginlik və nisbi uzanmasına təsiri nəzərdən keçirilmişdir. Onların pressformada soyudulmasının optimal şərtləri müəyyən olunmuşdur. Müxtəlif növ butadien-nitril kauçuklu dinamik vulkanlaşmış termoelastoplast nanokompozitlərin dağıdıcı gərginlik və nisbi uzanmaya təsiri öyrənilmişdir. Random polipropiləndən fərqli olaraq dinamik vulkanlaşmış nanokompozitlər onlar əsasında mürəkkəb konfigurasiyaya malik keyfiyyətli konstruksiya materialları almağa imkan verən izotrop xüsusiyyətlər ilə xarakterizə olunurlar.

Açar sözlər: termoelastoplast, butadien-nitril kauçuku, random polipropilen, bentonit, təzyiqlə altındakı tökmə.

ОСОБЕННОСТИ ЛИТЬЯ ПОД ДАВЛЕНИЕМ ДИНАМИЧЕСКИ ВУЛКАНИЗОВАННЫХ НАНОКОМПОЗИТОВ НА ОСНОВЕ РАНДОМ ПОЛИПРОПИЛЕНА, БУТАДИЕН-НИТРИЛЬНОГО КАУЧУКА И БЕНТОНИТА

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В работе рассмотрено влияние технологических параметров литья под давлением на основные физико-механические характеристики нанокompозитов динамически вулканизованных термоэластопластов на основе рандом полипропилена и бутадиен-нитрильного каучука различных марок. В качестве оборудования использовали одношнековый литьевой агрегат с 4-мя температурными зонами материального цилиндра. Вращательное и поступательное перемещение шнека позволяло осуществлять равномерное диспергирование смешиваемых компонентов смеси. Для улучшения совместимости полимерных компонентов в качестве компатибилизатора использовали привитой сополимер полипропилена с малеиновым ангидридом в количестве 1-3% масс. Изучено влияние температурного режима материального цилиндра литьевой машины и давления литья на разрушающее напряжение, относительное удлинение, модуль упругости на изгиб и объемную усадку. Показано, что увеличение указанных технологических параметров литья приводит к росту прочностных показателей, заметному снижению объемной усадки нанокompозитов динамически вулканизованных термоэластопластов. Рассмотрено влияние температуры прессформы и времени выдержки под давлением на разрушающее напряжение и относительное удлинение нанокompозитов. Установлены оптимальные условия их охлаждения в прессформе. Изучено влияние нанокompозитов динамически вулканизованных термоэластопластов с различными типами бутадиен-нитрильного каучука на разрушающее напряжение и относительное удлинение нанокompозитов. Показано, что в отличие от рандом полипропилена, динамически вулканизованные нанокompозиты характеризуются изотропными свойствами, позволяющими получать на их основе качественные конструкционные материалы сложной конфигурации.

Ключевые слова: термоэластопласт, бутадиен-нитрильный каучук, рандом полипропилен, бентонит, литье под давлением.