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RESEARCH ARTICLE

Study of the influence of ultra-high molecular weight polyethylene and high-density polyethylene on the properties and structure of ethylene propylene diene monomer rubber

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Abstract

Objectives. The study set out to examine the impact of pre-mixed ultra-high molecular weight polyethylene (UHMWPE) and high-density polyethylene (HDPE) on a range of properties and structural characteristics of SKEPT-50 ethylene propylene diene monomer (EPDM) rubber.

Methods. The production of rubber mixtures involved the pre-mixing of rubber with UHMWPE and HDPE in a Brabender PL 2200-3 plasti-corder chamber (Germany) at a temperature of 160°C, for a period of 6 min, and with a rotor speed of 60 rpm. The polyethylene constituents were incorporated into the rubber compound at concentrations of 5, 10, and 15 pts. wt. The subsequent introduction of the principal constituents of the rubber mixture was conducted in an SYM laboratory mill (China) for a period of 30 min at a temperature of no more than 100°C. The vulcanization of the samples was conducted in an Y1000D vacuum hydraulic press (China) at a temperature of 185°C for a period of 35 min. The investigation of vulcanization and physical and mechanical properties was conducted in accordance with the established protocols. The analysis of the rubber supramolecular structure was conducted using a JEOL JSM-6840 LV scanning electron microscope (Japan).

Results. The results demonstrate that an increase in the proportion of HDPE and UHMWPE to 15 pts. wt leads to a notable enhancement in the hardness of the rubbers by 10 and 5 Shore A units, respectively. The frost resistance coefficient at −45°C demonstrates an increase with the incorporation of 10 pts. wt of HDPE to reach a value of 0.229, and a further increase with the incorporation of 15 pts. wt of UHMWPE to reach a value of 0.260. The degree of swelling of rubbers in a DOT-4 brake fluid environment is observed to decrease to 13% for rubbers with HDPE and 19% with UHMWPE. The degree of swelling of rubbers in the DOT-4 brake fluid environment is observed to decrease to 13% for rubbers with HDPE and 19% with UHMWPE. While an increase in the HDPE content results in a 5% increase in volumetric wear, an increase in the UHMWPE content is associated with a 45% decrease in volumetric wear. The introduction of UHMWPE was observed to result in the formation of inclusions of varying shapes and sizes within a range of 50–100 µm. The transition zone between UHMWPE and rubber is characterized by a smooth surface. No evidence of cracks or micro-tears between the polymer phases, which could potentially form during low-temperature splitting, was observed. This finding indicates the presence of favorable interfacial interactions, which can be linked to the observed enhancements in resistance to aggressive liquids and abrasion, as well as the improved tensile frost resistance coefficient. The supramolecular structure of rubber samples combined with HDPE is more pronounced and exhibits greater relief than that of the original rubber. This is indicative of a more uniform distribution within the matrix volume, which can be attributed to the high fluidity of the HDPE melt.

Conclusions. Rubbers modified with UHMWPE, in comparison with HDPE, exhibit enhanced resistance to wear, oil, and frost, while maintaining their elastic and strength properties. It was established that rubber containing 15 pts. wt of UHMWPE exhibits optimal properties and can thus be recommended for use in sealing rubber products.

Keywords
ethylene propylene diene monomer rubber, rubber, high-density polyethylene, ultra-high molecular weight polyethylene, modification, physical and mechanical properties

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НАУЧНАЯ СТАТЬЯ

Исследование влияния сверхвысокомолекулярного полиэтилена и полиэтилена низкого давления на свойства и структуру резин на основе этиленпропилендиенового каучука

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Аннотация

Цели. Изучение влияния сверхвысокомолекулярного полиэтилена (СВМПЭ) и полиэтилена низкого давления (ПЭНД) на комплекс свойств и структуру резин на основе этиленпропилендиенового каучука марки СКЭПТ-50.

Методы. Резиновые смеси изготавливали путем предварительного смешения каучука с СВМПЭ и ПЭНД в камере пластикордера BRABENDER PL 2200-3 (Германия) при температуре 160°C в течение 6 мин и скорости вращения роторов 60 об/мин. Полиэтилены вводили в количестве 5, 10 и 15 мас. ч. на 100 мас. ч. каучука. Последующее введение основных ингредиентов резиновой смеси производилось на лабораторных вальцах SYM (Китай) в течение 30 мин при температуре не более 100°C. Вулканизацию образцов проводили в вакуумном гидравлическом прессе Y1000D (Китай) при температуре 185°C в течение 35 мин. Исследование вулканизационных и физико-механических свойств проведено стандартными методами. Исследование надмолекулярной структуры резин проведено с помощью сканирующего электронного микроскопа JEOL JSM-6840 LV (Япония).

Результаты. Показано, что с увеличением содержания ПЭНД и СВМПЭ до 15 мас. ч. твердость резин повышается на 10 и 5 единиц по Шору А соответственно. Коэффициент морозостойкости при −45°C увеличивается, достигая значений 0.229 при введении 10 мас. ч. ПЭНД и 0.260 при введении 15 мас. ч. СВМПЭ. Степень набухания резин в среде тормозной жидкости DOT-4 снижается до 13% у резин с ПЭНД и 19% со СВМПЭ. Исследование стойкости образцов резин к абразивному износу выявило различия в износостойкости в зависимости от вида термопласта: с увеличением содержания ПЭНД объемный износ повышается на 5% и снижается на 45% при увеличении содержания СВМПЭ. Исследования надмолекулярной структуры показали, что при введении СВМПЭ появляются включения разнообразной формы с размерами в пределах 50–100 мкм. Переходная зона между СВМПЭ и каучуком достаточно плавная, трещин и микроразрывов между фазами полимеров, которые могли бы образоваться в процессе низкотемпературного раскалывания, не наблюдается. Это свидетельствует об удовлетворительном межфазном взаимодействии и объясняет повышение стойкости к агрессивной жидкости и абразивному истиранию, а также увеличение коэффициента морозостойкости при растяжении. Образцы резин с ПЭНД по сравнению с исходной резиной имеют более выраженную и рельефную надмолекулярную структуру без видимых включений, что свидетельствует о более равномерном распределении в объеме матрицы за счет высокой текучести расплава ПЭНД.

Выводы. Резины, модифицированные СВМПЭ, по сравнению с ПЭНД обладают более высокими показателями износо-, масло- и морозостойкости при сохранении упруго-прочностных показателей. Установлено, что резина, содержащая 15 мас. ч. СВМПЭ, обладает наилучшим комплексом свойств и может быть рекомендована для использования в производстве уплотнительных резинотехнических изделий.

Ключевые слова
этиленпропилендиеновый каучук, резина, полиэтилен низкого давления, сверхвысокомолекулярный полиэтилен, модификация, физико-механические свойства

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INTRODUCTION

The principal focus of contemporary elastomer chemistry and technology concerns the modification of rubbers with additives to create elastomeric materials having enhanced characteristics to support a broader range of applications. One of the most effective methods for modifying the properties of rubbers is to combine them with thermoplastic polymers, including polyethylene, polypropylene, and polyvinyl chloride. The potential of thermoplastic polymers as modifying additives in rubber and rubber-based materials has been demonstrated in numerous studies by various authors [1–4]. Nevertheless, the problem continues to be of both scientific and practical interest due to the advancement of technologies for the production of novel thermoplastic and rubber materials and the potential for obtaining materials with defined properties.

As has been demonstrated in previous studies [1, 3], the enhancement of specific material properties during modification is contingent upon a high level of interaction at the elastomer–filler phase interface. This is particularly evident when a developed transition layer is formed at this interface, which depends on the compatibility of the polymers involved. Nevertheless, it remains a challenging endeavor to identify a compatible combination of high-molecular-weight rubber and polymer components for optimal distribution and interaction at the phase interface.

Due to their chemical resistance, high strength, operational viability across a wide temperature range, resilience to ozone, heat, atmospheric conditions, and frost, as well as market availability and relatively low cost [5–8], ethylene propylene diene monomer (EPDM) rubbers are of particular interest in the development of frost-resistant rubbers that can function effectively in the presence of aggressive environments. EPDM is an amorphous and nonpolar rubber that exhibits a specific affinity with thermoplastic polyolefins that possess analogous polarity and solubility parameters [7].

In this regard, the aim of the work is to study the effect of ultra-high molecular weight polyethylene (UHMWPE) and high-density polyethylene (HDPE) additions on the complex properties and structure of rubbers based on EPDM rubber of the SKEPT-50 brand. The selection is based on the fact that UHMWPE, which is distinguished by a considerable length of macromolecules with minimal branching, exhibits a lower degree of crystallinity, as well as offering high strength, wear resistance, frost resistance, resilience to shock loads, and a low friction coefficient [9–11]. Consequently, HDPE exhibits a high degree of density, hardness, and rigidity. It is important that the melting of HDPE and UHMWPE occurs in the same temperature range as the process of vulcanization of EPDM (130–150°C) [1].

MATERIALS AND METHODS

Triple ethylene propylene diene rubber of the SKEPT-50 brand produced by *Ufaorgsintez* (Russia) with mass fractions of propylene units 42–50% and dicyclopentadiene units 5.8–7.2% was used as the basis of rubber mixtures (TU 2294-087-05766563-2010). UHMWPE of the GUR 4113 brand with a medium-viscosity molecular weight of $3.9 \cdot 10^6$ g/mol produced by *Celanese* (Germany) and HDPE of the 273-83 brand with a medium-viscosity molecular weight of $0.5 \cdot 10^6$ g/mol produced by *Kazanorgsintez* (Russia) were selected as modifying additives. Polyethylene additions were introduced in amounts of 5, 10, and 15 pts. wt per 100 pts. wt of rubber. Formulations of rubber compounds based on SKEPT-50 are presented in Table 1. The following ingredients were also used to produce rubber mixtures: carbon black of the N550 brand produced by *Ivanovo Carbon Black and Rubber* (Russia) (CAS No. 1333-86-4), zinc oxide produced by *Chelyabinsk Chemical Plant "OXIDE"* (Russia) (CAS No. 1314-13-2), stearic acid produced by *Component-Reaktiv* (Russia) (CAS No. 57-11-4), altax produced by *Ningbo Actmix Rubber Chemicals Co.* (China) (CAS No. 120-78-5) and sulfur produced by *Kaspiygas* (Russia) (CAS No. 7704-34-9).

The rubber mixtures (2–7) are prepared by pre-mixing rubber with UHMWPE and HDPE in the Brabender PL 2200-3 plasti-corder chamber (*Brabender*, Germany) at a temperature of 160°C for 6 min and a rotor speed of 60 rpm. The subsequent introduction of the primary constituents of the rubber mixture was conducted on SYM laboratory rollers (*Yi Tzung*, China) for a period of 30 min at a temperature of no more than 100°C. The samples were then subjected to vulcanization in a vacuum hydraulic press Y1000D (*Tung Yu*, China) at a temperature of 185°C for a duration of 35 min. The optimal temperature and duration of vulcanization of rubber compounds were selected on the basis of the findings of studies into the vulcanization characteristics.

Table 1. Formulation of rubber compounds based on SKEPT-50

No.	Ingredients	Weight parts per 100 weight parts of rubber						
		1	2	3	4	5	6	7
1	SKEPT-50	100.0	100.0	100.0	100.0	100.0	100.0	100.0
2	HDPE 273-83	–	5.0	–	10.0	–	15.0	–
3	UHMWPE GUR 4113	–	–	5.0	–	10.0	–	15.0
4	Carbon black N550	50.0	50.0	50.0	50.0	50.0	50.0	50.0
5	Zinc oxide	5.0	5.0	5.0	5.0	5.0	5.0	5.0
6	Stearic acid	1.5	1.5	1.5	1.5	1.5	1.5	1.5
7	Altax	1.5	1.5	1.5	1.5	1.5	1.5	1.5
8	Sulfur	2.0	2.0	2.0	2.0	2.0	2.0	2.0

The vulcanization characteristics of rubber compounds were determined on a rotor-free analyzer for the recyclability of rubbers RPA 2000 from *Alpha Technologies* (USA) at a temperature of 185°C, a frequency of 1.7 Hz and a deformation amplitude of 0.5° for 50 min in accordance with GOST R 54547-2011[1]. The determination of physical and mechanical parameters, as well as the study of tensile frost resistance were carried out on the UTS-20K test machine (*UTS Testsysteme*, Germany) in accordance with GOST 270-75[2] and GOST 408-78[3], respectively. The resistance to ageing of the materials under the influence of static compression deformation was determined in accordance with GOST 9.029-74[4] at 20% compression and a temperature of 100°C. The wear resistance of the rubbers was evaluated by the method of determining abrasion resistance in accordance with GOST 23509-79[5] on an AR-40 friction machine (*Compart*, Russia). Shore A hardness was determined according to GOST 263-75[6]. The degree of swelling of vulcanizates in a medium of DOT-4 brake fluid (*LUXE*, Russia) was determined according to GOST 9.030-74[7]. The supramolecular structure of rubbers was studied using a JEOL JSM-6840 LV scanning electron microscope (*JEOL*, Japan) on low-temperature chips of rubber samples.

RESULTS AND DISCUSSION

Vulcanization characteristics of rubber compounds based on SKEPT-50 are shown in Table 2.

The study of the vulcanization kinetics of rubber compounds has shown that a decrease in the maximum torque (S'_{max}) as compared to the initial compound can be achieved by introducing modifying additives. An increase in the content of HDPE to 15 pts. wt leads to a decrease in S'_{max} by 29%, while an increase

[1] GOST R 54547-2011. National Standard of the Russian Federation. Rubber compounds. Measurement of vulcanization characteristics with the rotorless cure meters. Moscow: Standartinform; 2018.

[2] GOST 270-75. Interstate Standard. Rubber. Method of the determination elastic and tensile stress-strain properties. Moscow: Standartinform; 2008.

[3] GOST 408-78. State Standard of the USSR. Rubber. Methods for determination of low temperature resistance at extension. Moscow: Izdatelstvo standartov; 1985.

[4] GOST 9.029-74. State Standard of the USSR. Unified system of corrosion and ageing protection. Vulcanized rubbers. Method of testing of resistance to ageing under static deformation of compression. Moscow: Izdatelstvo standartov; 1982.

[5] GOST 23509-79. Interstate Standard. Rubber. Method for the determination of abrasion resistance under slipping a renewing surface. Moscow; IPK Izdatelstvo standartov; 2001.

[6] GOST 263-75. State Standard of the USSR. Rubber. Method for the determination of Shore A hardness. Moscow: Izdatelstvo standartov; 1989.

[7] GOST 9.030-74. Interstate Standard. Unified system of corrosion and ageing protection. Vulcanized rubbers. Method of testing of resistance to attack by corrosive media in limp state. Moscow: Standartinform; 2003.

Table 2. Vulcanization characteristics of rubber compounds

No.	Properties	Original rubber	5 pts. wt		10 pts. wt		15 pts. wt	
			HDPE	UHMWPE	HDPE	UHMWPE	HDPE	UHMWPE
		1	2	3	4	5	6	7
1	S'_{max}, dN·m	15.48	13.46	14.15	11.89	13.78	11.06	14.03
2	S'_{min}, dN·m	1.29	1.11	1.26	1.01	1.33	0.98	1.44
3	$S'_{max} - S'_{min}$, dN·m	14.19	12.35	12.89	10.88	12.43	10.08	12.59
4	T_5, min	2.13	2.24	2.24	2.28	2.23	2.28	2.22
5	T_{90}, min	18.78	17.74	17.49	18.13	17.80	18.58	18.38
6	R_V, min^{-1}	6.01	6.45	6.56	6.31	6.42	6.13	6.19

Note: S'_{max} is the maximum torque; S'_{min} is the minimum torque; $S'_{max} - S'_{min}$ is the torque difference; T_5 is the time of onset of scorching; T_{90} is the time to achieve optimum vulcanization; R_V is the vulcanization rate.

in the content of UHMWPE reduces it by 11%. The introduction of HDPE leads to a gradual decrease in the minimum torque (S'_{min}), which indicates a decrease in the viscosity of rubber mixtures [12–13] due to increased melt fluidity; conversely, the introduction of UHMWPE leads to an increase in the viscosity of rubber mixtures. This is due to the length of the polymer chains, which ensures transformation of UHMWPE into a highly elastic state rather than a viscous state upon heating [2, 14]. The difference between the maximum and minimum torque ($S'_{max} - S'_{min}$) characterizes the cross-linking density in rubber [15–16]. The maximum density of the cross-linking is recorded in the original rubber. The smallest difference is characterized by rubber compounds containing HDPE. This behavior of HDPE-filled rubbers can be explained in terms of the better distribution of HDPE in the elastomer matrix, which is achieved during the high-temperature mixing of rubber with HDPE powder due to the high fluidity of its melt to manifest a shielding effect of the rubber macromolecules from the sulfur vulcanizing system [2]. As the concentration of HDPE increases, this effect increases together with a decrease in the cross-link density. In turn, UHMWPE, which takes the form of micro-volumes in the volume of the matrix, additionally locally hinders the process of cross-linking of rubber macromolecules through sulfur bridges, which also leads to a slight decrease in $S'_{max} - S'_{min}$ as compared to the original rubber. In both cases, the introduction of the studied thermoplastics leads to an increase in the time of the mixtures in the viscous state (T_5) and the vulcanization rate (R_V), thus reducing the time for achieving optimum vulcanization (T_{90}).

Table 3 presents the physical and mechanical and low-temperature characteristics of vulcanizates based on SKEPT-50.

A study of the physical and mechanical properties of rubbers has shown that with the introduction of polyethylene and increase in its content, both the conditional stress at 100% elongation (f_{100}) and the Shore A hardness (H) of the vulcanizates increase. Compared to the original rubber, rubber materials with 15 pts. wt of HDPE and UHMWPE have f_{100} values that are 1.4 times higher and Shore A hardness that is 10 and 5 units higher, respectively. At the same time, the conditional tensile strength (f_{st}) and elongation at break (ε_e) values remain close to those of the original rubber.

Studies of the low-temperature properties of vulcanizates have shown that the maximum coefficient of frost resistance (K_{frost}) are at −45°C for rubbers containing UHMWPE: the higher the UHMWPE content, the higher the frost resistance value K_{frost}. This is due to the fact that, compared to HDPE, UHMWPE has a more developed amorphous region with interlacing of long macromolecules and through macromolecules (connecting crystallites), giving the material high elasticity and frost resistance. Therefore, it is likely that the amorphous phase of UHMWPE contributes to freeze resistance at low temperatures when the rubber macromolecules lose their flexibility [17]. Rubbers with HDPE (2, 6) have a frost resistance coefficient under tension of less than 0.20, which indicates insufficient frost resistance of the rubber at a given temperature.

It is known [18] that the resistance of rubber to aggressive media is primarily determined by the nature of

Table 3. Properties of vulcanizates based on SKEPT-50

No.	Properties	Original rubber	5 pts. wt		10 pts. wt		15 pts. wt	
			HDPE	UHMWPE	HDPE	UHMWPE	HDPE	UHMWPE
		1	2	3	4	5	6	7
1	f_{st}, MPa	18.1	17.5	18.9	17.7	16.8	19.6	18.6
2	f_{100}, MPa	2.8	3.2	3.0	3.3	3.3	3.8	3.8
3	ε_e, %	516	492	528	496	466	585	487
4	K_{frost} at −45°C	0.240	0.199	0.230	0.229	0.247	0.173	0.260
5	*H*, Shore A scale	62	65	63	67	66	72	67
6	RRCS (100°C × 24 h), %	52	51	37	54	53	51	51
7	Δ*M* in DOT-4 (100°C × 72 h), %	1.78	1.61	1.71	1.60	1.58	1.55	1.43

Note: f_{st} is the conditional tensile strength; f_{100} is the conditional stress at 100% elongation; ε_e is the relative elongation at break; K_{frost} is the coefficient of frost resistance during tension; *H* is the Shore A hardness; RRCS is the relative residual compressive strain at 20% compression; Δ*M* is the degree of swelling.

the rubber. At the same time, the components that make up rubber have a significant impact on the behavior of rubber materials in aggressive environments. Due to its chemical nature, EPDM has high resistance to the action of polar media and low resistance to aliphatic, aromatic, and nonpolar solvents [7, 19]. In order to assess the resistance of rubber to aggressive environments, we selected a polar glycol-based brake fluid DOT-4 Arctic Extreme manufactured by *LUXE* (Russia) for use in hydraulic brakes and clutches of cars with disc and drum braking systems. The results of the study showed that all rubbers of compositions 1–7 in the DOT-4 medium exhibited high resistance. Despite the low degree of cross-linking, the degree of swelling of the rubber decreases to 13% for HDPE and 19% for UHMWPE at increased concentrations of these additives. It is likely that the high molecular weights of the polymers contribute to a reduction in the degree of swelling.

An important characteristic that allows evaluating the relaxation properties and sealing ability of rubbers is the amount of relative residual compression strain (RRCS). For rubbers 2, 4–7, the RRCS values are at the level of the original rubber, while rubber 3, which contains 5 pts. wt UHMWPE, exhibits a decrease in RRCS up to 37%.

One of the approaches to reducing rubber wear and improving performance is to increase abrasion resistance. Figure 1 shows the results of the abrasion resistance measurements of the studied rubbers.

The study of the resistance of rubber samples to abrasive wear revealed differences in wear resistance

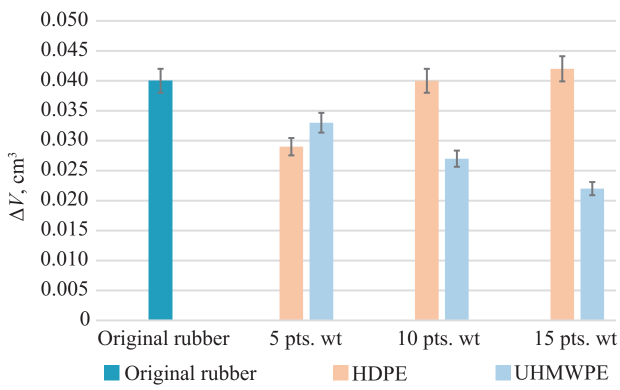

Fig. 1. Volumetric wear of rubber due to abrasive abrasion

depending on the type of polyethylene addition. When 5 pts. wt of both HDPE and UHMWPE were added to EPDM, volumetric wear was reduced by approximately 28% and 18%, respectively, as compared to the original rubber. The improved volumetric wear with increased HDPE content may additionally be due to a decrease in the cross-linking density of the rubber when HDPE is introduced. Increasing the UHMWPE content has a positive effect on the wear resistance of rubber. With the introduction of 15 pts. wt, volumetric wear is reduced by 45%. This is most likely due to the high tribotechnical properties of the UHMWPE itself.

Micrographs of low-temperature chips of rubbers containing modifying polyethylene additives obtained using a JEOL JSM-6840 LV scanning electron microscope are shown in Fig. 2.

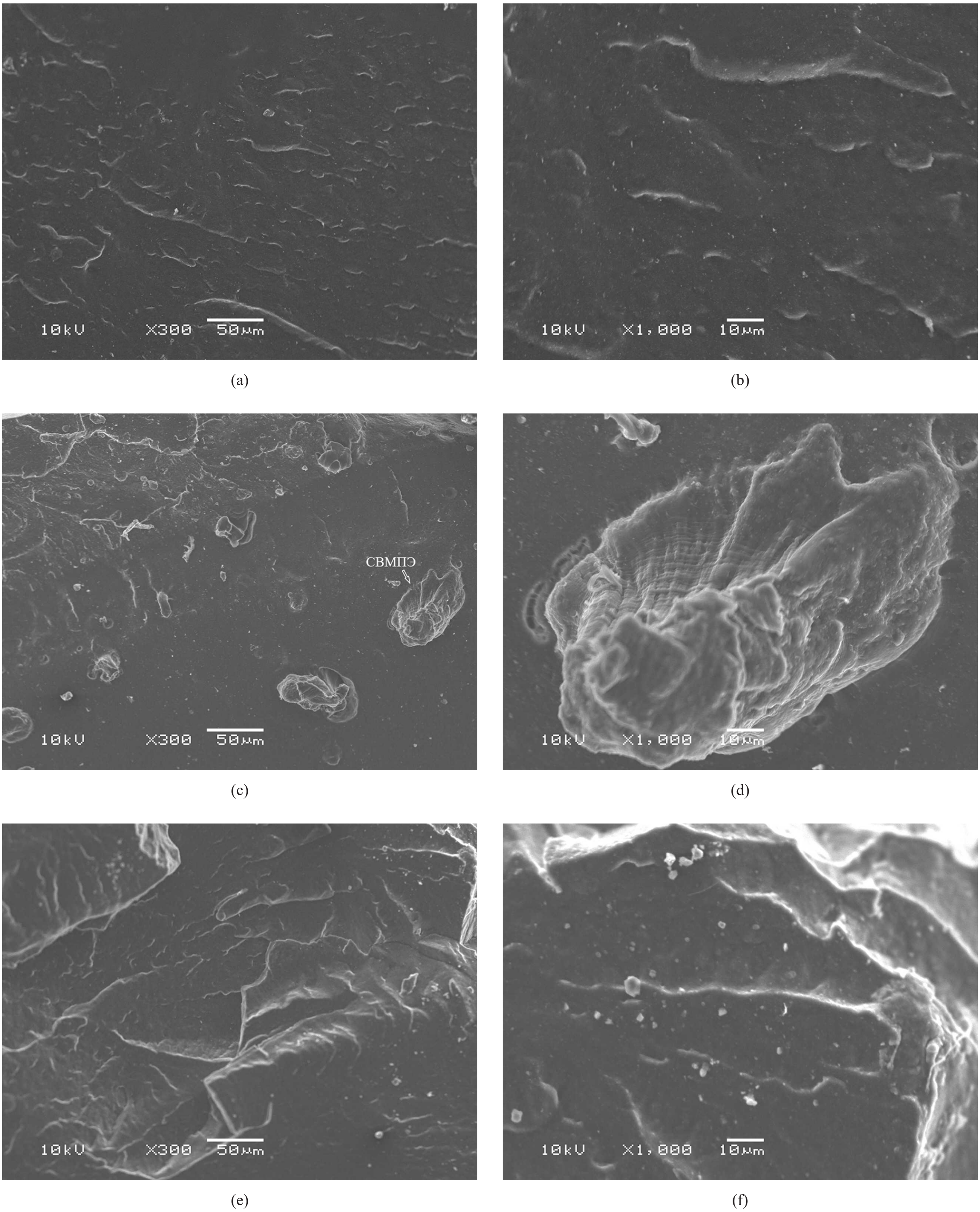

Fig. 2. Microphotographs of rubber based on SKEPT-50 (a, b); SKEPT-50 + 15 pts. wt of UHMWPE (c, d); SKEPT-50 + 15 pts. wt of HDPE (e, f) at magnifications 300× (left) and 1000× (right)

Figure 2 shows that the original rubber initially has a homogeneous structure (Figs. 2a, 2b). Following the introduction of UHMWPE, inclusions of various shapes with sizes ranging from 50–100 µm appear (Figs. 2c, 2d). In samples containing UHMWPE, the transition zone between UHMWPE and rubber appears quite smooth without any cracks or microfractures between the phases of polymers that could form during low-temperature splitting. This indicates satisfactory interfacial interaction to explain the increased resistance to aggressive liquids and abrasive wear, as well as an increase in the coefficient of frost resistance under tension [2]. The samples of HDPE rubber (Figs. 2e, 2f) have a more pronounced and embossed supramolecular structure as compared to the original rubber. The absence of visible inclusions as manifested in the UHMWPE samples indicates a more uniform distribution in the volume of the matrix due to the high fluidity of the HDPE melt.

CONCLUSIONS

A comparative assessment of the effect of HDPE 273-83 and UHMWPE GUR 4113 additions on the complex of technical properties of rubbers based on SKEPT-50 was carried out. UHMWPE modified rubbers, in comparison with HDPE, have higher wear, oil and frost resistance while maintaining elastic strength characteristics due to satisfactory interfacial interaction between UHMWPE and rubber. The rubber compound containing 15 pts. wt of UHMWPE had the best set of properties and can be recommended for use in the manufacture of sealing rubber products. This demonstrates the potential of using UHMWPE and HDPE as modifying additives to improve the performance of EPDM-based rubbers.

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Authors' contributions

M.D. Sokolova—guidance and scientific consulting, editing the release version of the article.

A.R. Khaldeeva—conducting experimental studies, processing and analyzing the data obtained, writing the text of the article, and preparing materials for publication.

M.L. Davydova—processing and analysis of the obtained data, editing the article text.

A.F. Fedorova—processing and analysis of the data obtained, editing the text of the article.

N.V. Shadrinov—management at all stages of work, editing the article text.

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