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To cite this article: L. Sudnik et al 2016 IOP Conf. Ser.: Mater. Sci. Eng. 153 012017

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The influence of nano-ceramic modifier on the structure and properties of polyolefin composites

L. Sudnik¹, V. Dubkova¹, A. Galinovsky², A. Osipkov²

¹ SNU Institute of Powder Metallurgy of National Academy of Sciences of Belarus, Platonova st., 41, Minsk, Belarus
² Bauman Moscow State Technical University, 2 Baumanskaya st., 5/1, Moscow, Russian Federation

osipkov@bmstu.ru

Abstract. It has been shown, that the nano-ceramic modifier boehmite, with specific surface area of up to 400 m²/g and dispersion degree of less than 100 nm, is a structurally active filler of an ultra-high-molecular-weight polyethylene. The boehmite influences the polymer crystalline and supra-molecular structure, during crystallization of the polymer from the melt under uniaxial plastic deformation conditions. It results in the production of nano-composites with an improved complex of properties.

1. Introduction

Nowadays, composite materials based on polyolefins are high on the list of thermoplastic materials that are filled and enhanced by various modifiers. The most popular in various technical fields and national economy is ultra-high-molecular-weight polyethylene (UHMWPE) [1, 2]. UHMWPE is distinct from conventional polyethylene in that its molecule consists of hundreds of thousands of units, and the resulting molecular mass is about few millions (> 1.5 x 10⁶ g/mol). Super high molecular mass of this polymer determines its unique properties, which are vastly different from the other types of polyethylene [3–6]. UHMWPE has improved physical and mechanical properties, corrosive and chemical resistance, cold-resistance, extremely high impact resilience, hardness and dimensional stability, working efficiency in a wide temperature range, and resistance to abrasion and gamma-ray radiation. UHMWPE is used in conditions where traditional types of polyethylene and many other polymers do not withstand the severe operating conditions. UHMWPE may be used as an alternative of the other more expensive materials (steel, bronze, polymeric amide, fluorine plastic, etc.) and may be technically required, i.e. being the only material with the capability for this purpose.

Industrially, for the first time the UHMWPE was used in 1950s by the Ruhrchemie AG company. In the 1960s AJ Pennings, in co-operation with the DSM company (Netherlands), synthesized fibre structures with extremely high strength from UHMWPE [7, 8]. Currently UHMWPE is produced using various catalysts by a number of manufacturing companies in Japan, USA and Europe; such as Farbwerte Hoechst Ruhrchemie, Ticona (Germany), Mitsui Chemicals Group (Japan), Koninklijke Philips N.V. (Netherlands), [9]. Manufacturing of UHMWPE in the Russian Federation is mastered by PJSC Kazanorgsintez and LLC Tomskiy neftekhimicheskiy zavod.

The properties of the polymer produced depend on the composition of the catalysts and on the ethylene polymerization conditions at these catalysts. Depending on the field of application and
UHMWPE processing methods, various powder types of this polymer are used, which are distinguished by molecular mass, particle size and supramolecular structure. By making appropriate choices, it is possible to change persistently the functional properties of the UHMWPE, and expand the fields of application its composites. Thus, one of the ways to improve the mechanical characteristics of UHMWPE is the addition into polymer of short basaltic fibres or dispersive components as the micro- and nano-particles (kaolinite, carbon nano-tubes, ceramics) [10, 11]. Much attention is given to the development of composite materials based on the UHMWPE with improved tribo-technical properties for operation under dry friction and abrasive wear conditions [12–15].

2. Objective of the work
In the references mentioned above, the functional additives in the UHMWPE compositions are used in limited amounts. The amount of added modifiers is predominantly of 0.5 – 20 % by mass (with a maximum of 40 % by mass) [16]; however, under the conditions of ever higher demand for polymeric composite materials, and the ever increasing scarcity and high cost of the raw material resources that are necessary for polymer manufacture, the objective to reduce the polymeric fraction in compositions while preserving or increasing the specified technical and operational characteristics, is relevant.

The objective of the present investigation is the development and examination of the polyolefin composite properties based on the ultra-high-molecular-weight polyethylene (UHMWPE) and nano-ceramic modifier with increased content of UHMWPE.

3. Experiment
The reactor powder of UHMWPE, produced by LLC Tomskiy neftekhimicheskiy zavod, was used as the object of investigation. The average polymer molecular mass was greater than 4.0х10^6 g/mol. The nano-ceramic modifier chosen was the nano-crystalline aluminium monohydroxide (boehmite), with specific surface up to 400 m²/g, dispersion degree less than 100 nm. The nano-crystalline aluminium monohydroxide (boehmite), with a specific surface of up to 400 m²/g and dispersion degree of less than 100 nm, was used as the nano-ceramic modifier. The boehmite was produced by hydrothermal synthesis, in the Belarusian State Research and Production Concern of Powder Metallurgy [17]. Polymeric nano-composites were produced by means of powder technology: dry blend compositions using mechanical activator were pre-cooked, then the blend compositions were mono-lithiated by the method of compression moulding at optimal temperature and time conditions. To compare data results correctly, a UHMWPE sample without the modifying additive was produced, under the same conditions of pre-cooking and moulding.

To examine the structure and properties of the composite, the following methods were used: scanning electron microscopy, electro-physical, acoustic, X-ray phase and thermo-mechanical analyses. X-ray analysis of the boehmite, unfilled UHMWPE, and its compositions with the boehmite after moulding, was carried out on the DRON-3 diffractometer using CuKα-irradiation. Primary processing of the diffraction data obtained, and phase identification of the samples of crystalline compound mixture, were performed using the software packages Powder X and WinXpow [18], and the radiographic powder standards data bank JCPDS PDF2 [19]. Crystallite size analysis was carried out by means of “Size/Strain” software, in order to determine the sizes of coherent scattering region (CSR) and microstresses. In both cases, the calculations are based on integral width variation of diffraction maxima of a crystalline compound with respect to the corresponding line values lying in the same ranges of 2q for a standard substance. Here, q is the Bragg angle for diffraction peaks, and the quantity 2q is used to determine the inter-planar distances.

A silicon wafer was used as a standard. The degree of crystallinity in the samples was determined by the X-ray diffraction method and using Crystallinity software. The calculations in the software are performed by least square method for all points obtained from the three X-ray diffraction patterns, including the error caused by the air scattering. The patterns include the X-ray diffraction pattern of
the substance under investigation and the X-ray diffraction patterns of compounds with known crystallinity indices, which have diffraction maxima in the same 2q region.

Thermo-mechanical investigations of samples, with diameter and height of 10 mm and exactly parallel planes, were performed under heating with constant rate (5 deg/min) using a Hoppler consistometer, modernized for this purpose, under conditions of uniaxial compression and constant pressure of 0.6 MPa. Electro-physical characteristics were determined by means of a E7-14 LCR meter on the operating frequency of 100 Hz, with the error no more than 0.1%. The dynamic modulus of elasticity of the composite was calculated based on the speed of ultrasound transmission through the sample. The hardness of the composite samples was determined on the Hoppler consistometer with the vertex angle of a hardened steel cone of 53° + 10′ on the samples. All sample tests were performed at room temperature.

Morphology of the fractures of UHMWPE compositions with the boehmite was researched by means of a Mira scanning electron microscope with a high resolution, made by Tescan ORSAY HOLDING, a.s. (Czech Republic). The elemental composition was determined by means of energy-dispersive X-ray microanalysis on an INCA 350 test machine, Oxford Instruments (England).

The thermomechanical researches for the moulded samples of UHMWPE, and its compositions with the boehmite, was determined according to GOST 15088-83 [20]. The essence of the method is the determination of the temperature at which a standard indenter penetrates to a depth of 1 mm, under the action of force, into the test sample heated with the constant speed.

A burning test of a moulded samples of both unfilled and boehmite filled UHMWPE was performed according to GOST 28157-89, Method b [21].

4. Research results
X-ray analysis of tableted UHMWPE, and its compositions with the boehmite, showed that the major crystalline phase of UHMWPE ordered regions in all of the samples is β-polyethylene, which is crystallized in orthorhombic syngony. Along with the crystal peaks of the orthorhombic symmetry group, the peaks on the X-ray diagrams with a lower intensity, related to the monoclinic lattice, were also observed.

![Crystallinity degree of UHMWPE on the boehmite content in moulded composites](image)

**Figure 1.** Dependence of X-ray crystallinity degree of UHMWPE on the boehmite content in moulded composites.
Even weak mechanical impacts onto the UHMWPE are shown to disarrange its crystalline structure. The X-ray observed degree of crystallinity is negligibly decreased (up to 1.8 %). It was observed that the monoclinic syngony content increased following mechanical impact; however, after moulding, these changes are levelled, and the monoclinic syngony in unfilled monolithiated UHMWPE is practically non-existent. On the addition of small amounts of the boehmite into the UHMWPE, the monoclinic syngony of UHMWPE crystalline phase is observed again, and the X-ray polymer crystallinity degree is increased. At the same time, this dependence has an extremum (figure 1).

An increase in the dimensions of the crystallite coherent scattering regions (CSR), for the compositions with the boehmite is observed after pressing of the mixed powder structures. At the same time, the crystallites sizes are increased with the degree of increased boehmite in the composition; however, the micro-stresses in the crystalline lattice ($\Delta d/d \times 10^{-3}$) are decreased (table 1). The CSR dimension increase of nano-modifier after UHMWPE, with the boehmite powder composition structures moulding, is also observed properly.

| Boehmite content, % by mass. | CSR dimensions, Å | $\Delta d / d \times 10^{-3}$, kg/mm$^2$ |
|-----------------------------|-------------------|-----------------------------------------|
|                             | Bohmite | UHMWPE | Bohmite | UHMWPE |
| -                           | -       | 252.4  | -       | 5.87    |
| 5                           | 171.6   | 276.9  | 5.40    | 5.40    |
| 70                          | 455.0   | 328.2  | 2.77    | 4.49    |

It is noteworthy, that in the X-ray diffraction patterns of the moulded composites of the boehmite with the UHMWPE, new reflections are observed. These reflections point to the structural transformations of the boehmite when composition processing. The reflections are visualized on the fullest extent at mild nano-modifier contents. For instance, an addition of 5 % by mass of boehmite into the UHMWPE composition and mono-lithiating of the composition, results in the observation of reflections related to the crystalline boehmite phase and reflections relating to oxide and aluminium hydroxide. The proportion of the crystalline phases is 54: 35: 11 % by mass respectively. When increasing the boehmite content in the composition to 70 % by mass, the boehmite phase is increased and the percentage proportion is 87: 9: 4 % by mass respectively.

Investigations of the supramolecular structure of the various compositions by electron microscopy showed the following results. On the spalled sample of the moulded unfilled UHMWPE, spots of a prolate spherilitic structures with the length up to dozens μm are observed. The spherulites are oriented by their extended sides in the plane perpendicular to the pressure direction during moulding, see figure 2(a). The spherulites are distributed uniformly enough on the entire spalled spot surface and interstratified with amorphous (disarranged) component, figure 2(b), concentrated at the outer surfaces of the polycrystalline aggregates. When UHMWPE crystallizes from the melt with the boehmite under uniaxial plastic deformation conditions, the general character of the morphological formation is not changed. A formation of spherulites also takes place; however, there is a tendency to its increasing, figure 2(c). The nano-modifier was displaced in an amorphous polymeric phase, incorporated in its supramolecular structures, which are almost perpendicular located to the surface of the crystallites, and tended to regularize, figure 2(d). The observed structuring of the composite were also indicated by increased ultrasonic velocity in the nano-composite sample.
Figure 2. SEM images of (a, b) unfilled UHMWPE; and UHMWPE composition of (c, d) 5% boehmite by mass, and (e, f) 70% boehmite by mass.

X-ray microanalysis (figure 3) confirms the elemental composition in chosen points of the morphological polymeric forms.

It is seen, figure 3(a), that for the unfilled UHMWPE, on the various levels of supra-molecular configuration, the spectra are characterized only by presence of carbon, hydrogen and oxygen. In the analogous spectra for supra-molecular structures of UHMWPE composition with the nano-dispersed boehmite, aluminium along with the above elements is well-marked and incorporated into the supra-molecular formations, figure 3(b).

The results of SEM investigation, X-ray diffraction and X-ray spectroscopic analyses show an active influence of the boehmite on the formation of macro- and micro-structures of the polyolefin composite and agree satisfactorily with our research results of a physical and chemical interaction the boehmite and the UHMWPE described before [22].
Concerning the physical, mechanical and deformational properties nano-composites produced, it is necessary to mark the following aspects. For addition of up to 40% by mass of boehmite to the UHMWPE, a reinforcing effect is not observed. The hardness, dynamic elastic modulus and thermal stability of the composites are decreased (table 2, 3). A significant decrease in the temperatures of the phase transitions and the relative deformation increase are observed on the thermos-mechanical curves for the UHMWPE composites with a low boehmite content (figure 4, curve 2). Two regions of α-relaxation, caused by segmental mobility of the chains in a disarranged (amorphous) polymer region, are well marked. The first one is caused by the segmental mobility in the adsorbed (boundary) polymer layer, for which the structure differs from the composition volume. The second point of inflexion corresponds to the characteristic temperature region for the unfilled UHMWPE (figure 4, curve 1). For the addition of ≥ 50% by mass of nano-ceramic modifier to the polymer a dramatic change of the polymer properties is observed. All the physical and technical properties of the UHMWPE composition with the boehmite are improved (table 2, 3).

The composition of UHMWPE with 70% by mass of boehmite was tested for resistance to burning under torch flame, according to the GOST 28157-89 Method b standard [21]. The tests showed that

| Spectrum  | C, O, H % by mass | Al % by mass | Spectrum  | C, O, H % by mass | Al % by mass |
|-----------|------------------|-------------|-----------|------------------|-------------|
| Spectrum 1| 100              | 0.00        | Spectrum 1| rest             | 6.82        |
| Spectrum 2| 100              | 0.00        | Spectrum 2| rest             | 6.72        |
| Spectrum 3| 100              | 0.00        | Spectrum 3| rest             | 8.43        |
| Spectrum 4| 100              | 0.00        | Spectrum 4| rest             | 6.91        |
| Spectrum 5| 100              | 0.00        | Spectrum 5| rest             | 8.74        |
| Spectrum 6| 100              | 0.00        | Spectrum 6| rest             | 5.02        |
| Spectrum 7| rest             | 7.31        |           |                  |             |

Figure 3. SEM micro-images and X-ray microanalysis results of (a) the unfilled UHMWPE, and (b) UHMWPE with 5% boehmite mass.
the sample placed in the torch flame is carbonized and did not emit smoke. There is no dropping. For the unfilled UHMWPE, there was intensive smoke emission and dropping of melted and burning particles of material was observed.

![Thermo-mechanical curves of moulded UHMWPE](image)

**Figure 4.** Thermo-mechanical curves of moulded UHMWPE
(1) unfilled, (2) containing 5 % by mass of boehmite and (3) containing 70 % by mass of boehmite.

| Boehmite % by mass | Density, kg/m³ | Hardness, MPa | Dynamic elastic modulus, GPa | % Relative deformation at temperature of 220 °C |
|-------------------|----------------|---------------|-----------------------------|-----------------------------------------------|
| 0.0               | 1120           | 145           | 5.2                         | 8.0                                           |
| 5.0               | 1130           | 145           | 5.1                         | 22.0                                          |
| 30.0              | 1150           | 150           | 4.7                         | 21.0                                          |
| 40.0              | 1230           | 160           | 3.0                         | 20.0                                          |
| 50.0              | 1380           | 220           | 6.3                         | 6.0                                           |
| 70.0              | 1570           | 240           | 6.9                         | 3.0                                           |
Table 3. Physical and technical characteristics of composition based on UHMWPE and boehmite.

| Boehmite, % by mass | Dielectric relaxation time at frequency of 100 Hz, $\tau_p$, $[s \times 10^6]$ | Vicat thermal stability, °C |
|---------------------|---------------------------------------------------------------------------------|-----------------------------|
| 0.0                 | 37.3                                                                             | 230                         |
| 5.0                 | 9.7                                                                              | 175                         |
| 30.0                | 19.5                                                                             | 180                         |
| 40.0                | 44.3                                                                             | 190                         |
| 50.0                | 65.9                                                                             | 290                         |
| 70.0                | 165.8                                                                            | 300                         |

5. Discussion
The experimental results show that for UHMWPE with high volume fractions of boehmite, there is an observed ambiguous change in the properties of the material produced. It may be explained from the perspective of induced interfacial layer. It is known that the structure and properties of polymer composite materials are determined by a number of factors, that is: the structure and properties of its individual components, its mixing conditions and compositions formation, interfacial phenomena, supra-molecular structure and the induced interfacial layer. At the same time, the processes, which take place in the interphase boundary of filler-polymer, are dominant in composite materials properties formation. The fundamentals of the theory of interfacial phenomena in polymer composite materials were laid down since the last century by YuS Lipatov and his school [23], and are still relevant. When the UHMWPE composition is processed with the presence of investigated nano-ceramic filler, some of the macro-molecules, mostly high-molecular fractions, pass into the boundary layers and interact with the polymer [24]. The volume of the polymer is enriched by lower molecular fractions with an increased mobility. It is also known, that the addition of a filler in the rigid chain polymer leads to a reduction of macro-molecule packing density in the boundary layers. As a result, an additional free volume is create, and at sufficiently high volume fractions a looser polymer structure with an increased mobility of the chain components is formed.

The data mentioned above for the thermos-mechanical analysis and the acoustic and electro-physical measurement indicate that for UHMWPE with low volume fractions of boehmite there is still an insufficiently ordered structure. For boehmite concentrations of up to 40 % by mass, a reduction of the dielectric relaxation periods and dynamic elasticity modulus is observed (table 3). The deformation properties of the composites under heating conditions are reduced (table 2, figure 4, curve 2). The physical and mechanical properties and the thermal stability are either reduced or remain at the level of that of the unfilled polymer. The observed properties are not exceptional and accord with the thermoplastic composite materials consisting of a limited amount of excipient and reinforcement, previously developed by the present authors [25, 26]. In general, it is typical for many thermoplastic materials with a low content of dispersed fillers. As for the nano-ceramic modifier boehmite used in this work, along with its many advantages it has a significant disadvantage: the fact that boehmite is not a reinforcing filler this limits its usage in polymeric compositions [27].

In this work it was shown that for UHMWPE with the addition of ≥ 50 % by mass of boehmite, when practically all the polymer passes into the interfacial and surface layers, a significant decrease in the mobility of the macro-molecules is observed, as suggested by the dielectric relaxation periods (table 3). The thermos-mechanical analysis data also confirm this hypothesis, see figure 4, curve 3, where the curve trajectory is sharply changed. A significant decrease (of between 1.3 and 2.7 times) of the deformation of the polymeric composite is observed in the whole temperature range studied, and an increase of the thermal stability up to between 290 and 300 °C (table 2, 3) was also observed. Under high volume fraction filling of UHMWPE by boehmite, a completely-bound system is formed,
resulting from the physical and chemical transformations in the inter-phase boundary, proceeds with a simultaneous formation of a physical network of boehmite within the composition volume (figure 2e, f). This results in an improvement of all the technical characteristics of the UHMWPE composition. The research results are in satisfactory agreement with the fundamental concepts of the formation of polymer structures in the presence of fillers described in the monograph [28].

6. Conclusions
New highly filled polyolefin composite materials based on UHMWPE and nano-ceramic modifier (boehmite) were developed. It was established that the boehmite actively influences the physical and chemical processes, which take place in the contact region of the nano-ceramic filler – ultrahigh molecular weight polyethylene, under the conditions of composite formation. A correlation of the processes which proceed in interfacial layer, with the structure and properties of the materials produced for various fill fractions of boehmite in the polymer was determined. A sharp increase of the mechanical and thermal properties of the composites is observed for fill fractions of the UHMWPE by the boehmite of equal or greater than 50 % by mass, and reaches a maximum at 70 % boehmite content, when practically the whole polymer is situated on the surface layer in the form of the very thin films. A significant decrease of the macro-molecule mobility in this layer, in conjunction with the formation of a physical network of the active reinforcement within the composition volume, leads to the formation of a completely-bound polymer structure within composition material. As a consequence, the density of the composite of UHMWPE filled with 50 – 70 % by mass of boehmite is increased by 1.2–1.4 times, the dynamic elastic modulus by 1.2–1.3 times, the hardness by 1.5–1.7 times, and the thermal stability by 60–70°C.

These increased physical, mechanical and thermal characteristics (non-halogenated fire retardant) allow the highly filled composites based on the UHMWPE and the boehmite to be considered for functional designation for workpieces and details of the constructions used in various fields of science, technics and industry. For instance, high requirements for burnability and environmental friendliness are specified for modern materials used in electronic devices constructions and electro-technics. Valid prescriptions, including EU directive of the limiting usage dangerous materials, prohibit the usage of additives with bromine and chlorine content. Taking into account the increased level of fire-resistance of the UHMWPE boehmite composite, this material is able to replace the polymeric compositions used before in mentioned above technical fields.

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