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# UHMWPE-based nanocomposite as a material for damaged cartilage replacement



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#### ABSTRACT

In the present work dispersion-strengthened nanocomposites based on ultra-high molecular weight polyethylene (UHMWPE) after mechanical activation were studied. Mechanical activation was performed for hardening of the boundaries between the polymer particles, reducing the fusion defects and increasing of wear-resistance. Three types of samples were prepared: UHMWPE, UHMWPE/Al<sub>2</sub>O<sub>3</sub> nanocomposite and UHMWPE/Al<sub>2</sub>O<sub>3</sub> nanocomposite after mechanical activation. UHMWPE/Al<sub>2</sub>O<sub>3</sub> nanocomposites prepared with mechanical activation show the best mechanical properties in compression and higher wear-resistance. UHMWPE/Al<sub>2</sub>O<sub>3</sub> nanocomposites prepared with mechanical activation were chosen for in vivo study by orthotopical transplantation in rats. Animals' activity has been being monitored for 60 days after surgery. No signs of inflammation, cellular infiltration, destruction of material or bone–cartilage defect were found. Implanted sample has not changed its position of implantation, there were no any shifts. Obtained data shows that UHMWPE-based nanocomposite is a promising material for creating bioimplants for cartilage defect replacement.

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#### 1. Introduction

Problem of articular cartilage repair is one of the main challenges in medicine. Diseases of bones and cartilage are very common and they are one of the main reasons of lowering life quality [1–3]. Hyaline cartilage, which covers articular surfaces of the bones, has very poor repair capabilities, because it has no blood vessels, has anaerobic metabolism, dense matrix, and few cells, which cannot migrate. Cartilage lesions, which do not reach the subchondral bone, cannot be repaired [3]. If the lesion had reached the subchondral bone, then natural regeneration starts. This regeneration is provided by fibrous cartilage and it does not provide restoration of functions [1,2]. Also some authors suggest that defect usually grows bigger after damage and the tissue on the edge of defect degrades, and this may degrade the joint functioning [4].

None of existing methods can provide a complete restoration of defect. There is no authentic data about the restoration of defects with chondroprotective drugs which block apoptosis of chondrocytes and supply cells with cartilage matrix components [5]. Surgery methods are more perspective. It is possible to find many studies with good or satisfactory results after applying different surgery methods [1–4]. For example, microfracture method provides regeneration of fibrous cartilage. But this method is good for repair only of small defects, because it does not provide complete restoration of joint function [2]. A more perspective method is using of scaffold to repair the defect. For this approach different types of grafts can be used — allografts, autografts, and synthetic materials

[2]. Autograft is considered as gold standard for defect replacement, because it provides histocompatibility and integration without rejection [1], but this method has many limitations: limited size of defect, it needs several surgeries, and uncertain condition of donor zone. All these reasons make applying of this approach impossible in some cases [1,2]. Alternative method is to use biocompatible polymer implants, which allows avoiding limitations of autografts or allografts. One of the most perspective materials is ultra high molecular weight polyethylene (UHMWPE) [6,7].

UHMWPE as a material for joint prostheses is studied quite extensively [8–10]. The biggest problem is the stability of UHMWPE implants under load while using in a human body and retention of mechanical and tribological properties. In many studies, the goal of which was to increase the wear resistance, a method of radiation crosslinking and heat treatment was used [11–13]. However, radiation may lead to an accelerated oxidative degradation of UHMWPE and change of the mechanical properties [14–17]. In the present work, dispersion-strengthened UHMWPE-nanocomposites after mechanical activation in planetary ball mill for hardening of the boundaries between the polymer particles, reducing the fusion defects and wear were studied.

#### 2. Materials and methods

#### 2.1. Preparation of test specimens

The UHMWPE powder (JSC "Kazanorgsintez",  $2\times 10^6$  g/mol) was used as the polymer matrix. Al $_2O_3$  nanopowder (JSC "Siberian Chemical Combine", average particle size of 20 nm) was used as bioinert filler.

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Three types of samples were prepared: UHMWPE, UHMWPE/ $Al_2O_3$  composite and UHMWPE/ $Al_2O_3$  composite after mechanical activation (MA). The degree of filling was 3%  $Al_2O_3$  by weight, since a greater degree of filling may lead to the formation of dispersed particle monolayer on the surface of the polymer particles covering the full surface of the polymeric powder. As a result, communication between the individual powder particles can be extremely low [18].

Mixing of UHMWPE and 3% weight  $Al_2O_3$  nanopowder without mechanical activation was carried out in a mill IKA M20 (IKA-Werke GmbH & Co. KG, Germany) for 5 min at 20,000 rev/min.

Mechanical activation of UHMWPE and 3% wt. Al<sub>2</sub>O<sub>3</sub> nanopowder was performed in a planetary ball mill Fritsch Pulverisette 5 (Fritsch GmbH, Germany), equipped with agate bowls (500 ml) and corundum grinding balls 10 mm in diameter to minimize penetration of impurities. Filling of bowls with grinding bodies was about 45%. Bowls in the form of a cylinder filled with the material and balls, were mounted vertically. The motor drives the planet carrier, and a holder entrain in a circular motion bowls around the center of the carrier, as well as around its own axis. Rotational speed of the carrier was 370 rev/min. Then mechanically activated powder was pressed under a load of 70 MPa at 160 °C for 50 min and cooled under pressure.

Samples of different shapes were prepared: cylindrical, dumbbell-shaped and hemisphere with a diameter of 1.8 mm. Samples of the material, which showed better mechanical and tribological properties were used to prepare samples for testing in vivo (hemisphere with a diameter of 1.8 mm).

#### 2.2. Study of fracture surface

The surface of the three types of samples (UHMWPE, UHMWPE/Al $_2O_3$  composite and UHMWPE/Al $_2O_3$  composite after mechanical activation) after tensile tests was studied by scanning electron microscopy (SEM). SEM was performed with Hitachi TM-1000 (accelerating voltage 5 kV). For studying of non-conductive polymer samples the surface of UHMWPE and UHMWPE-based composite samples was covered with a layer of platinum (10–20 nm) by Auto Fine Coater JFC-1600 (Jeol, USA).

#### 2.3. Study of mechanical and tribological properties

Mechanical testing was performed on a universal testing machine Zwick/Roell Z 020, (Zwick GmbH & Co. KG, Germany) using cylindrical samples  $24 \times 12.5$  mm for compressive tests (ASTM D695) and dumbbell-shaped samples (type IV) for tensile tests (ASTM D638).

The tribological tests were carried out in the mode of friction in distilled water by pin-on-disk method on CETR-UMT-3 (Bruker AXS, Switzerland) with normal load 50 N, counter-body: steel 45; sliding velocity 200 rpm, length of friction 4 km, and  $T=37\,^{\circ}$ C. These parameters were similar to standard parameters of cartilage friction tests in some other works [19–21].

#### 2.4. Study of biocompatibility

All experiments with animals were performed according to ethic requirements. Wistar male rats  $(200{\text -}400~\text{g})$  were used as model (n=5). Rats were narcotized with aether. After that an incision on the knee and baring of knee ligament was done. Then it was cut and the knee joint was bent. After that articular surfaces of the knee became visible and a 1.8 mm hemispheric defect in tibia articular surface was made with a drill. The sample was placed into the created defect. After that surgery wounds were sutured and rats were put into a vivarium for 60 days.

After 60 days of keeping rats in a vivarium, they have been euthanized and samples have been taken out for further study. The knee joints were studied macroscopically and after that histological slides, stained with hematoxylin–eosin, have been made to be studied with a microscope.

#### 3. Results

#### 3.1. Mechanical properties

Samples in tensile tests showed a different type of destruction. Ductile fracture is observed in the case of UHMWPE, which is confirmed by SEM (Fig. 1A). Introduction of the filler leads to the embrittlement of the material, and brittle fracture is observed (Fig. 1B). Processing of powder components by mechanical activation resulted in the change of the nature of the destruction as shown in Fig. 1C.

Comparative tests of cylindrical specimens (UHMWPE, UHMWPE/ ${\rm Al}_2{\rm O}_3$  nanocomposite and UHMWPE/ ${\rm Al}_2{\rm O}_3$  nanocomposite after mechanical activation) were made to estimate the influence of the applied technology of material processing on the mechanical properties of dispersion-strengthened nanocomposites. Evaluation was carried out in two ways: by measuring the Young's modulus in compression, which determines the elasticity of the material, and wear resistance of the fixed specimen on a rotating disk to identify the tribological behavior of the material.

Results of mechanical testing in compression are illustrated in Fig. 2. Elastic modulus of UHMWPE filled with alumina after mechanical activation (MA) was the highest.

Fig. 3 demonstrates tribological properties of 3 tested samples. After 1 h of friction the wear rate (Z) of UHMWPE, UHMWPE/Al<sub>2</sub>O<sub>3</sub> nanocomposite and UHMWPE/Al<sub>2</sub>O<sub>3</sub> nanocomposite after mechanical activation accorded 0.13, 0.23, and 0.05 mm, respectively.

#### 3.2. Study of biological properties in vivo

Rats' activity was monitored in a vivarium with standard conditions during 60 days after surgery. Rats moved free in cages and used all limbs for moving. On the 14th day there was no lameness, surgery wounds disappeared without any consequences, and rats behaved like before the surgery. The ioints were studied macroscopically at the moment of taking out. No signs of inflammation and purulence were found. Lengthwise section of the bone became visible after embedding the bone into paraffin and making slides. Tight integration of sample into the bone without shifts was observed (Fig. 4). There was no further destruction of tissues around the defect. Size of the defect had not increased and the rest of the bone remained unchanged.

Study of histological slides also showed biocompatibility of the sample. Articular surface which was not damaged had its natural state. The structure of the sample was not damaged or changed; the surface of the sample, which was adjacent to the bone, was smooth, without any lesions and flaws. Inflammation or rejection was not caused. Fig. 5 demonstrates that the sample is adjacent tightly to the defect, and the defect is filled with material and there are no tissues of the organism in the defect. Sample remains in the defect and has not undergone destruction and it acts as a physical barrier for tissues, which has not grown into the defect. This barrier prevents overgrowth of connective tissue. Healthy tissues surround the sample with no signs of destruction around the defect. Newly formed cartilage tissue (Fig. 7) and bone tissue (Fig. 5) are observed on the microphotographs. As it can be seen on microphotograph (Fig. 6), the amount of connective tissue is small; it contains blood vessels; material contacts it tightly, without any gaps; the surface of the sample is plain without any cracks and traces of resorption. Contact with cartilage tissue is tight, without gaps, as it is shown in Fig. 7. Combination of intact sample without destruction of the material, without resorption and the absence of tissue destruction around the sample provided tight integration of the sample without movements. There were no signs of cellular infiltration near the zone of implantation, which can be considered as the absence of acute inflammation.

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