ISSN 0208-7774 TRIBOLOGIA 6/2018 p. 137–148

Sergiy SHEYKIN*, Irina POHRELYUK, Ihor ROSTOTSKYI*, Dmytro IEFROSININ*, Waldemar TUSZYŃSKI***, Anita MAŃKOWSKA-SNOPCZYŃSKA*****

TRIBOLOGICAL BEHAVIOR OF THE FRICTION PAIR "GRADE 2/PE-UHMW" AND THE TECHNOLOGY OF THE PRODUCTION OF ITS SPHERICAL PART MADE OF GRADE 2

WŁAŚCIWOŚCI TRIBOLOGICZNE BIOMATERIAŁÓW TYPU „GRADE 2/PE-UHMW" ORAZ TECHNOLOGIA WYTWARZANIA GŁÓWKI ENDOPROTEZY ZE STOPU GRADE 2

Key words: endoprosthesis, titanium alloy, friction pair, contact pressures, roughness, accuracy, friction, wear.

Abstract An advance in the material engineering of biomaterials intended for hip joint replacement prostheses is a very important social matter due to incurable diseases or injuries of human joints happening to many people. For this purpose, a development of the tribological test techniques is also an urgent issue.

The paper is devoted to the study of the tribological behaviour of the new "Nitrided GRADE 2/PE-UHMW" friction pair intended for hip joint endoprostheses. A pendulum tribometer and a ring-on-plane machine were used for tribological research.

A new lubricant based on chondroitin sulphate was tribologically investigated compared to commonly used bovine blood serum. Also the technology of thermo-diffusion nitriding (TDN) of the GRADE 2 femoral head was optimised based on the results of the tribological experiments. A means of selecting of the geometrical parameters of the prosthetic implants to achieve the smallest wear is discussed. The optimum technology of machining of the femoral head made of the GRADE 2 titanium alloy to obtain the best surface quality is also presented.

The paper consists of the first part of the results of the Polish-Ukrainian cooperation in testing biomaterials. The second part, to be also submitted to *Tribologia*, will describe the aspects of methodology of endoprosthesis simulation tests and test results, and it will present a difference in the wear of the hip joint prosthesis – the developed and commercial one..

Słowa kluczowe: stop tytanu, endoproteza, para trąca, naprężenia kontaktowe, chropowatość, dokładność wykonania, tarcie, zużycie.

Streszczenie Postęp w inżynierii materiałowej biomateriałów stosowanych w endoprotezach stawu biodrowego jest bardzo ważną sprawą społeczną z powodu nieuleczalnych chorób lub urazów stawów ludzkich, które zdarzają się wielu osobom. W tym celu pilną kwestią jest także rozwój tribologicznych technik badawczych.

Artykuł poświęcony jest badaniu właściwości tribologicznych nowej pary trącej nazwanej „Nitrided GRADE 2/PE-UHMW", przeznaczonej na endoprotezy stawu biodrowego. Do badań tribologicznych wykorzystano tribometr wahadłowy i maszynę typu pierścień–płaszczyzna.

Nowy smar na bazie siarczanu chondroityny został poddany badaniom tribologicznym i porównany do powszechnie stosowanej surowicy krwi bydlęcej. Również technologia azotowania termodyfuzyjnego (TDN) główki endoprotezy (GRADE 2) została zoptymalizowana w oparciu o wyniki eksperymentów tribologicznych. Omówiono sposób doboru parametrów geometrycznych implantów protetycznych w celu osiągnięcia najmniejszego zużycia. Przedstawiono również optymalną technologię obróbki głowicy endoprotezy wykonanej ze stopu tytanu (GRADE 2) w celu uzyskania najlepszej jakości powierzchni.

Artykuł zawiera pierwszą część wyników uzyskanych w ramach polsko-ukraińskiej współpracy w zakresie badania biomateriałów. W drugiej części, która również jest planowana do publikacji w czasopiśmie Tribologia, zostaną opisane aspekty metodologii testów symulacyjnych endoprotez i wyniki badań oraz przedstawiona różnica w zużyciu endoprotezy stawu biodrowego – opracowanej i handlowej.

* Bakul Institute for Superhard Materials – NAS of Ukraine, Avtozavodska str., 2, Kiev 04074, Ukraine.
** Karpenko Physico-Mechanical Institute – NAS of Ukraine, Scientific str., 5, Lviv 79000, Ukraine.
*** Institute for Sustainable Technologies – National Research Institute, Tribology Department, ul. Pulaskiego 6/10, 26-600 Radom, Poland.

INTRODUCTION

Endoprosthetics of the human joints is an effective method of the restoration of the working capacity of a person in the case of incurable diseases or injuries. Today, the most common operation of bone surgery is a hip joint replacement. Annually, the prosthetics of the hip joint is required for 500–1000 patients per 1 million people [**L. 1**]. For every 3–4 primary operations, one revision is conducted.

The reason for the surgical revision operation can be negative phenomena associated with the insufficient biocompatibility of the applied materials with the tissues of the human body, loosening of the stem in the bone, wear of the elements of the endoprosthesis leading to its instability, etc. It is obvious that the durability of the endoprosthesis is the main criterion of its quality and is determined to a large extent by the service properties of the materials used, an important place among which is the wear resistance and tribological characteristics. Thus, studies in this direction are extremely relevant.

It should be noted that a material that fully meets all the requirements has not been created to date. Each material has its advantages and disadvantagesm which are described in sufficient detail in the literature, e.g., [**L. 2, 3**].

The articulation (friction pair) is the most important part of the endoprosthesis that determines its durability. In 1958 Charnley [**L. 4**] identified the problem of creating a friction pair of the endoprosthesis as primarily a problem of a tribological nature. The researcher suggested using CoCrMo alloy for the femoral head material and ultrahigh molecular weight polyethylene (PE-UHMW) for the acetabulum (liner) – **Fig. 1**.

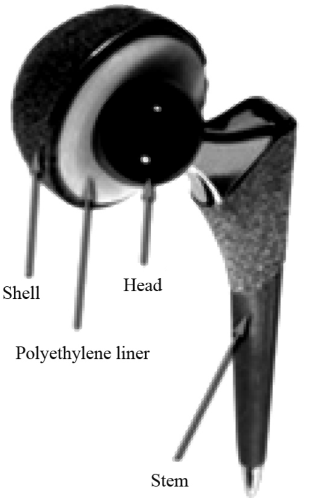

Fig. 1. General view of the hip joint replacement prosthesis [L. 4]
Rys. 1. Widok ogólny endoprotezy stawu biodrowego [**L. 4**]

The articulation with such a combination of materials has received the name of the gold standard of endoprosthetics and is still the most widely used in orthopaedic practice. According to a survey carried out in 35 hospitals in the USA (from 1999 to 2007), the CoCrMo / PE-UHMW endoprosthesis are used in 55% cases, while metal-on-metal and ceramic-on-ceramic in only 37 and 6%, respectively [**L. 4**]. Such a joint can remain operative for 20 years or more. According to [**L. 2**], the reserves of the wear resistance of the PE-UHMW-metal pair have not been exhausted at this point. However, it should be noted that, when used in the clinical practice, cases of granulomatous inflammation caused by the products of wear of PE-UHMW are documented [**L. 3**].

Today, various materials and their combinations are used for manufacturing the components of the replacement prosthesis. For femoral heads, the CoCrMo alloy, various types of ceramics, and less often titanium alloys are used, and for the acetabulum, PE-UHMW, CoCrMo alloy, and ceramics are used.

A significant share in the total volume is occupied by combinations of metal / PE-UHMW and ceramics / PE-UHMW. For the manufacturing of femoral heads, metals, i.e. CoCrMo alloy and titanium alloy GRADE 5, are normally used [**L. 2, 4**]. It is necessary to explain here that the term "GRADE" relates to the classification of titanium alloys, and it is used by many metal traders all over the world.

However, from the point of view of biocompatibility, these materials are not the best [**L. 5**] – **Fig. 2**. In the figure, the relative corrosion resistance is expressed as a percentage of ideal biocompatibility, where GRADE 2 corresponds to ≈ 99%. The corrosion resistance is strictly related to biocompatibility with the human body, and low corrosion resistance can cause the release of carcinogens into living tissue and blood.

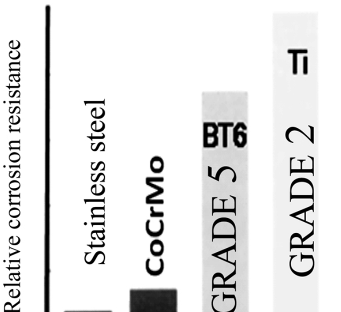

Fig. 2. Biological compatibility of various materials used for femoral heads [L. 5]
Rys. 2. Biokompatybilność rozmaitych materiałów używanych na główki endoprotez [**L. 5**]

Apparently, from the biological point of view, among metals and alloys, the technically pure titanium is the best for manufacturing the endoprosthesis heads [**L. 1, 2, 4–12**]. However, its main drawbacks, which hinder its use in endoprosthetics, are poor mechanical and tribological characteristics. This does not allow the use of parts made of commercially pure titanium for manufacturing the components of endoprosthesis without modification of the working surface, which should result in an optimal combination of mechanical and tribological characteristics. Such a combination is possible by using nitriding as the modification method [**L. 13, 14**]. In our opinion, the technology of thermo-diffusion nitriding (TDN) should be used to modify the working surface of such a critical part of the endoprosthesis as the femoral head. The method has advantages over others, e.g., the presence of a transitional diffuse layer between the thin surface layer of Ti-N compounds and the base material eliminates lapping and also ensures 100% reproducibility of the material features [**L. 15**].

TRIBOLOGICAL TEST METHODS

The choice of working fluid for tribological testing and technological regimes of TDN were made based on the results of studies performed using a pendulum tribometer (**Fig. 3**) and a ring-on-plane machine (**Fig. 4**).

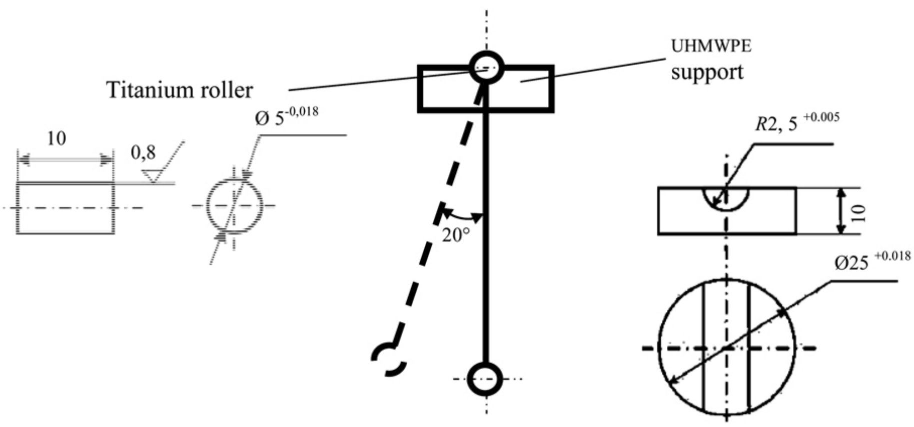

Fig. 3. The tribosystem of a pendulum tribometer
Rys. 3. Węzeł tarcia tribometru wahadłowego

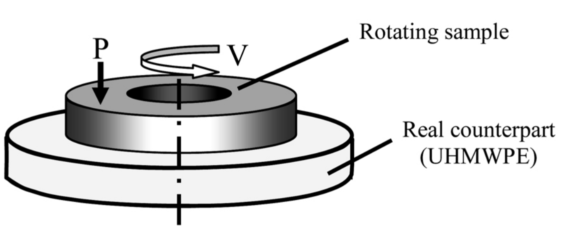

Fig. 4. The ring-on-plane tribosystem
Rys. 4. Węzeł tarcia typu pierścień–płaszczyzna

The length of the pendulum (**Fig. 3**) was 0.51 m, the weight of the load was 35.67 N, and the initial deviation angle was 20°. The coefficient of friction was estimated from Equation (1) [**L. 16**]:

$$f = \frac{\Delta A}{4(n-1)r} \quad (1)$$

where: f – coefficient of friction; ΔA – decrease in amplitude of oscillations of the pendulum for a period [m]; r – radius of the roller of the friction support unit [m], n – number of oscillation cycles.

During the tests using the ring-on-plane machine (**Fig. 4**), the rotating sample in the form of a ring was pressed against a stationary PE-UHMW disk. The friction force and the normal force were measured with a strain gauge dynamometer. By profiling, the cross-section area of the wear groove was determined. The sliding speed of the metal sample was v = 0.057 m/s and the contact pressure q = 3.54 MPa. The roughness of the working surface of the metallic sample was Ra = 0.8 μm, and for the PE-UHMW disk (ISO 5834-2), Ra was 3 μm.

The tribological characteristics of the friction pair were estimated from the coefficient of friction f and the wear intensity I of the surgical component, expressed in [mm^3/km].

It must be kept in mind, however, that it is not always correct to determine the contact pressure by dividing the load by the contact area. Each interaction scheme can have its own characteristics, which leads to uneven distribution of normal stresses along the contact surface. Considering that the endoprosthesis friction unit should have a very high degree of reliability, it is necessary to make sure that this approach is correct. For this purpose, the pressure in the contact zone was calculated using a dedicated software package.

As a result, it was found that the uneven distribution of the contact pressure along the friction surface of the ring does not exceed 3%, which, in our case, is quite legitimate to determine the normal stresses on the contact surface by dividing the load by the area of the contact surface.

RESULTS AND DISCUSSION

Aspects of selection of the working fluid

To obtain the correct results, it is necessary to strive to bring the conditions of the laboratory tests as close as possible to those of the friction in the natural joint. According to the classical provisions of tribology, the environment in which the rubbing parts interact largely determines the functional characteristics of the friction pair. According to **[L. 17]**, a third body forms between the surfaces of the rubbing bodies, whose properties are largely determined by the working medium. An ideal option would be to perform articulation tests in human synovial fluid, but it is impossible to obtain it in the volume necessary for the tests. According to ASTM F732-82, in the pin-on-plate tests, serum of bovine blood should be used as a working fluid. This is due to the fact that the synovial fluid is a blood serum (BS) transudate, i.e., they contain the same components which determine the frictional conditions in the human joint. Minor differences in their compositions affecting the friction conditions are practically negligible [**L. 16**].

The tribological function of the synovial fluid is realized due to the presence of liquid-crystalline components (LC) as complex esters of cholesterol acids. LCs form on the friction surfaces and orientate as a structure consisting of a number of nematic layers, the intermolecular interaction of which is small. This structure is similar to the structure of layered solid lubricants. During friction, the shear is localized between the layers, providing low friction [**L. 16, 18**]. It was found that the introduction of LC into the working liquid leads to a significant reduction in the friction of virtually all materials used in practice [**L. 13**]. Thus, the use of the bovine blood synovial fluid (SF) as a working medium in the tribological tests of artificial joints makes it possible to obtain the most reliable information about its functional characteristics.

However, the disadvantage of the SF is the time-limited use. When tested at 37°C, it quickly loses its properties, which can cause errors and, in addition, can lead to clogging of the pipelines of the testing equipment. Taking this into account, as well as the fact that tests in simulator stands require a significant volume of the working fluid, the question of replacing SF with a working fluid that is adequate in terms of tribological properties and can maintain its properties during tests performed for a longer time is a topical task.

In [**L. 19**], the results of tribological studies, using a pendulum tribometer and a drug containing chondroitin sulphate, which is part of the synovial fluid and performs a lubricating function, are presented. In the studies, joints of animals were used. As a result it has been established that the fluid containing chondroitin sulphate and natural synovium are approximately equal in tribological efficiency – **Fig. 5**.

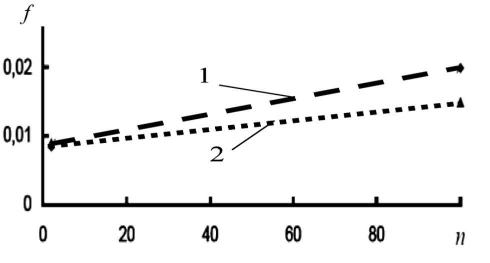

Fig. 5. Dependence of the friction coefficient *f* of the natural cartilage-cartilage pair on the number of oscillations of the pendulum tribometer when lubricated by: 1 – fluid containing chondroitin sulphate, 2 – SF [L. 16].

Rys. 5. Zmiana współczynnika tarcia *f* pary trących się naturalnych chrząstek stawowych wraz z rosnącą liczbą cykli oscylacyjnych tribometru wahadłowego przy smarowaniu z użyciem: 1 – cieczy zawierającej siarczan chondroityny, 2 – cieczy synowialnej SF [**L. 16**]

Taking this into account, in order to study the possibility of replacing BS in tribological tests of medical friction pairs, studies were conducted on the tribological efficiency of the drug "Artiflex-chondro" (AC) produced by LLC Pharmaceutical Company "Zdorovie" in Kharkov, Ukraine. The drug is a 10% aqueous solution of chondroitin sulphate, produced from animal raw materials.

In the tests on the pendulum tribometer, the components of the GRADE 2 titanium friction pair were used, whose surface was modified by TDN technology in various modes, which provided different surface microhardnesses: 12.6, 6.0, 5.5, and 5.2 GPa.

In the tests using the ring-on-plane machine, the following materials were used: a CoCrMo sample (HV 4.5 GPa), samples of GRADE 2 modified by ion-plasma thermocyclic nitriding (IPTN) (surface microhardness HV 6 GPa), and TDN (surface microhardness HV 4.9 GPa) [**L. 13**].

The results of the tests of the AC drug using the pendulum tribometer and the ring-on-plane machine are shown in **Figs. 6** and **7,** respectively.

It can be seen that the value of the friction coefficient (f) when using the drug AC and BS differ slightly. In testing using the pendulum tribometer, the maximum difference in f (14%) occurs when the titanium sample with a surface hardness of 12.6 GPa is used (**Fig. 6**). Using the ring-on-plane machine, the differences in the values of the friction coefficient and the wear rate of the

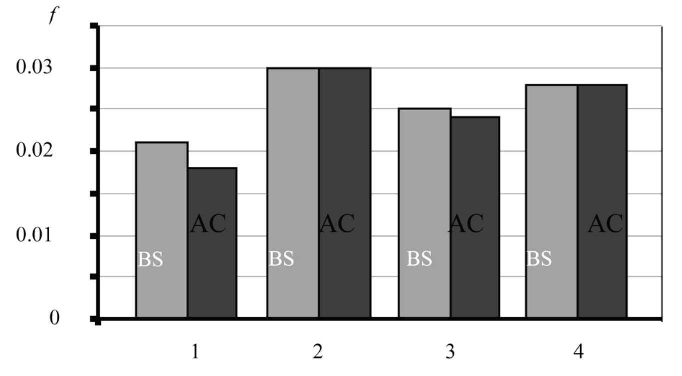

Fig. 6. Coefficients of friction *f* during testing of pairs "GRADE 2 (TDN)/PE-UHMW," lubricated with different media, using the pendulum tribometer: 1 – "GRADE 2 (HV 12.64 GPa)/PE-UHMW", 2 – "GRADE 2 (HV 6.0 GPa)/ PE-UHMW," 3 – "GRADE 2 (HV 5.5 GPa)/PE-UHMW," 4 – "GRADE 2 (HV 5.2 GPa)/PE-UHMW"; BS – blood serum, AC – Artiflex Chondro

Rys. 6. Współczynnik tarcia *f* otrzymany w czasie badania różnych skojarzeń materiałowych typu "GRADE 2 (TDN)/PE-UHMW", smarowanych dwiema cieczami, z użyciem tribometru wahadłowego: 1 – "GRADE 2 (HV 12.64 GPa)/PE-UHMW", 2 – "GRADE 2 (HV 6.0 GPa)/PE-UHMW", 3 – "GRADE 2 (HV 5.5 GPa)/PE-UHMW", 4 – "GRADE 2 (HV 5.2 GPa)/ PE-UHMW"; BS – serum z krwi, AC – Artiflex Chondro

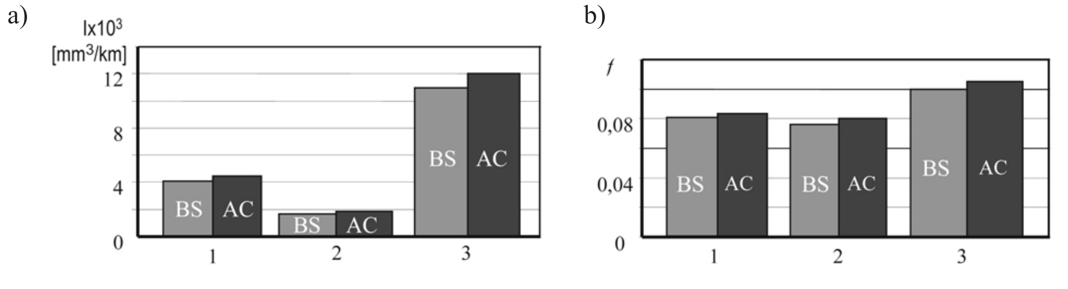

Fig. 7. Wear intensity *I* (a) and friction coefficient *f* (b) during testing of pairs lubricated with different media, using the ring-on-plane machine: 1 – "GRADE 2 (IPTN)/PE-UHMW," 2 – "GRADE 2 (TDN)/PE-UHMW," 3 – CoCrMo/PE-UHMW; BS – blood serum, AC – Artiflex Chondro.

Rys. 7. Wskaźnik zużycia *I* (a) i współczynnik tarcia *f* (b) otrzymany w czasie badania różnych skojarzeń materiałowych smarowanych dwiema cieczami, z użyciem tribometru typu pierścień-płaszczyzna: 1 – "GRADE 2 (IPTN)/PE-UHMW", 2 – "GRADE 2 (TDN) / PE-UHMW", 3 – CoCrMo/PE-UHMW; BS – serum z krwi, AC – Artiflex Chondro

surgical component lubricated with the BS and AC do not exceed 5% and 4%, respectively (**Fig. 7**).

Laboratory tests show that the AC product retains its properties in tribological tests 3 to 4 times longer than BS.

The differences in the values of the friction coefficient in the tests using the two methods can be a consequence of a significant difference, both in the dynamics of the tribometers and in the conditions of friction.

Thus, the AC product can be successfully used in studies of tribological characteristics of friction pairs "CoCrMo / PE-UHMW" and "Nitrided GRADE 2 / PE-UHMW" using bench tribometers and simulator stands.

Aspects of selection of the technological regime of TDN

The aim of the tribological research was also to establish an optimal thermal diffusion nitriding (TDN) technological regime [**L. 15**], providing the highest functional characteristics of the friction pair "GRADE 2/ PE-UHMW" by comparing with such characteristics of other friction pairs. The TDN method makes it possible to saturate the surface with nitrogen ions. This causes a change of the physico-mechanical properties of the material surface layer; in this case it is the titanium alloy (GRADE 2).

Table 1 shows the properties of the GRADE 2 samples after application of 16 different TDN regimes (which altered the temperature-time and gas-dynamic saturation parameters): surface roughness, surface microhardness, the wear intensity of the PE-UHMW component, and friction coefficient. For comparison, under the same conditions, samples from the CoCrMo alloy (*HV* 4.5 GPa) and ZrO_2 ceramics [**L. 14**] were also tested.

Table 1. TDN regimes, surface properties, wear intensity (*I*) of the PE-UHMW component, and friction coefficient (*f*) of the pair "Nitrided GRADE 2/PE-UHMW"

Tabela 1. Parametry azotowania termodyfuzyjnego (TDN), właściwości powierzchni, wskaźnik zużycia próbki z PE-UHMW (*I*) oraz współczynnik tarcia (*f*) skojarzenia meriałowego „Nitrided GRADE 2/PE-UHMW"

No.	TDN regime	*Ra* [µm]	$H_{0.49}$ [GPa]	*I* [mm³/km]	*f*
R1	850°C, 8 h, p_{N2} = 105 Pa	1.50	18.8	3.20	0.10
R2	950°C, 8 h, p_{N2} = 105 Pa	6.56	22.5	–	0.38
R3	850°C, 6 h, p_{N2} = 1 Pa	–	10.7	2.80	0.22
R4	900°C, 4 h, p_{N2} = 1 Pa	–	8.9	1.00	0.10
R5	650°C, 5 h, p_{N2} = 105 Pa	0.10	4.5	0.59	0.12
R6	650°C, 20 h, p_{N2} = 105 Pa	0.10	8.7	5.46	0.22
R7	700°C, 5 h, p_{N2} = 105 Pa	0.11	7.5	2.60	0.21
R8	700°C, 20 h, p_{N2} = 105 Pa	0.17	12.6	2.26	0.16
R9	750°C, 5 h, p_{N2} = 105 Pa	0.13	12.0	0.35	0.08
R10	750°C, 20 h, p_{N2} = 105 Pa	0.33	17.0	0.33	0.07
R11	750°C, 10 h, p_{N2} = 1 Pa	0.13	8.2	0.57	0.08
R12	750°C, 20 h, p_{N2} = 105 Pa + 750°C, 4 h, 1 mPa	0.31	15.2	0.30	0.07
R13	750°C, 20 h, p_{N2} = 105 Pa + 750°C, 6 h, 1 mPa	0.29	12.3	0.56	0.10
R14	750°C, 20 h + 800°C, p_{N2} = 105 Pa	0.25	10.2	0.38	0.08
R15	650°C, 20 h + 800°C, p_{N2} = 105 Pa	0.37	16.7	0.22	0.06
R16	650°C, 20 h + 800°C, 0,5h, p_{N2} = 105 Pa	0.18	9.0	1.06	0.07

As can be seen from **Table 1**, the best results are exhibited by a sample that was nitrided in the R15 regime (nitriding in molecular nitrogen atmospheric pressure: heating to 650°C, annealing for 20 hours, heating to 800°C, cooling in a stove). **Fig. 8** shows the results obtained in the comparative tests of this pair and the "Stainless steel/PE-UHMW," "CoCrMo/PE-UHMW," and "ZrO_2/PE-UHMW" pairs.

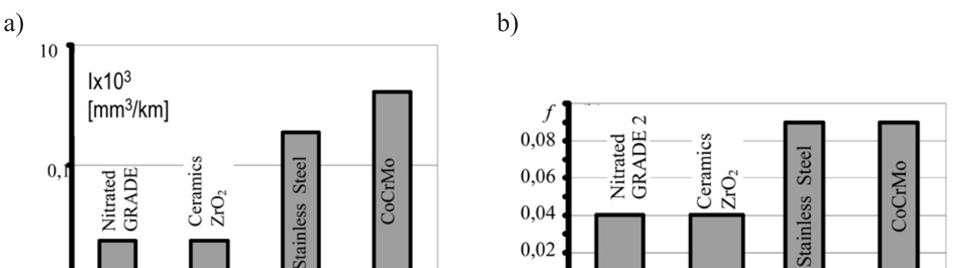

Fig. 8. Wear intensity of *I* (a) and friction coefficient *f* (b) during testing of various materials rubbing against PE-UHMW sample; research performed using the ring-on-plane machine

Rys. 8. Wskaźnik zużycia *I* (a) i współczynnik tarcia *f* (b) otrzymany w czasie badania różnych materiałów skojarzonych z PE-UHMW; badania wykonane za pomocą maszyny typu pierścień–płaszczyzna

It can be seen that nitrided GRADE 2, i.e. modified by TDN, can compete with ceramic analogues (for example, ZrO_2) on the same level, being several times better than the CoCrMo metal analogue [**L. 13**]. In this case, the fragile destruction of the titanium component is completely ruled out. Characteristically, after passing 200 km of friction, which corresponds to 23 million cycles of loading, the wear of the titanium component was not detected.

Aspects of hip joint endoprosthesis design

The intensity of wear of hinged joints is largely determined by the load (contact pressure). To determine the main factors that influence the contact pressure in the joint, the finite element calculation for the case of a femoral (spherical) head from GRADE 2 and an acetabulum from PE-UHMW has been performed. The problem was solved using dedicated software [**L. 20**]. **Fig. 9** shows a diagram of the contact pressure in the joint at zero gap. The diagram shows that the largest values of the contact pressure are located along the axis of symmetry.

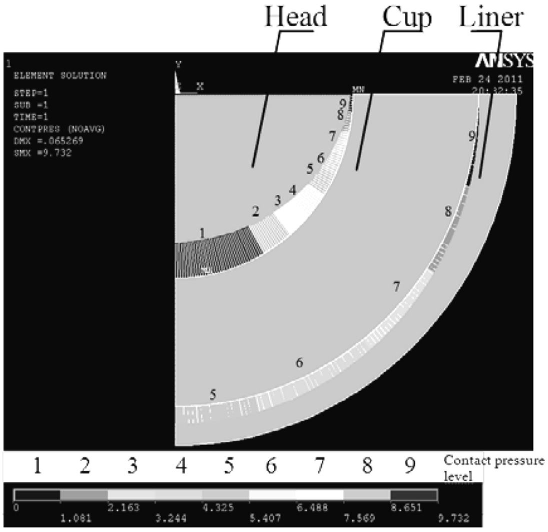

Fig. 9. Distribution of contact pressures between the elements of the endoprosthesis: 1 – femoral head; 2 – acetabulum; 3 – clip

Rys. 9. Rozkład naprężeń kontaktowych pomiędzy elementami endoprotezy: 1 – główka; 2 – panewka; 3 – zacisk

The dependence of the contact pressure on the forces and gap, which is well approximated by the static function (98% correlation), is obtained using Equation (2):

$$\sigma_{\max}(\delta, P) = a_1 + a_2 P^{a_3} + a_4 \delta^{a_5} + a_6 P^{a_3} \delta^{a_5} \quad (2)$$

where: $\sigma_{\max}(\delta, P)$ – maximum contact pressure [MPa]; P – load [N]; δ – radial clearance [mm], a_1, a_2, ... – coefficient of approximation function [MPa], the values of which are given in **Tab. 2**, depending on the diameter of the femoral head.

Maximum contact pressure at zero gap is determined using Equation (3):

$$\sigma_{\max}(\delta, P) = 1.72 \frac{P}{D^2} \quad (3)$$

where: $\sigma_{\max}(\delta, P)$ – maximum contact pressure [MPa]; P – load [N]; D – diameter of the sphere [mm]; 1.72 – coefficient obtained by statistical processing of data. The error of approximation of the calculated data is not more than 1%.

Table 2. Coefficients of approximation function [MPa]

Tabela 2. Współczynniki funkcji aproksymacyjnej [MPa]

D [mm]	a_1 [MPa]	a_2	a_3	a_4	a_5	a_6
28	–0.90849	0.01122	0.81321	13.92142	0.72374	0.018412
38	–0.57037	0.00823	0.77824	8.19921	0.70792	0.017300
48	–0.53733	0.01074	0.69556	5.01997	0.67063	0.025249
58	–0.48997	0.01032	0.66045	3.78452	0.65636	0.025285

The analysis of Equations (2) and (3) shows that the increase in the diameter of the contact surfaces of the joints from 28 to 58 mm (ca. 2 times) leads to a decrease in the contact pressure by more than 4 times, and the increase in the gap leads to an increase in the contact pressure.

Considering that the endoprosthesis should have a very high level of the reliability, the tests were also carried out for the "Nitrided GRADE 2/PE-UHMW" pair at higher contact pressures. The results are shown in **Fig. 10**.

It can be seen that the increase of the contact pressure to values of ~6.5 MPa does not lead to a noticeable change in the wear intensity of the polyethylene sample. However, for high values of the contact pressure, the wear becomes catastrophic, which, in our opinion, is explained by the plasticity of the PE-UHMW.

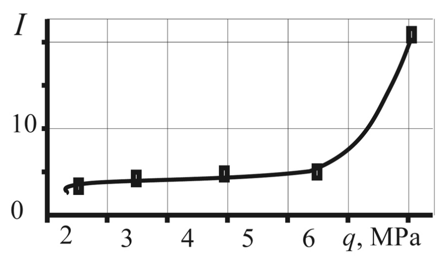

Fig. 10. Dependence of the wear intensity (*I*) of PE-UHMW on the contact pressure (*q*) in pairs with the nitrided sample GRADE 2; research performed using the ring-on-plane machine

Rys. 10. Zależność intensywności zużywania polimeru PE-UHMW od naprężeń kontaktowych *q* przy tarciu o próbkę azotowaną GRADE 2; badania wykonane za pomocą maszyny typu pierścień–płaszczyzna

Based on the results obtained, one can formulate some practical recommendations regarding the choice of the size of the joint for a particular patient and the manufacturing of joints. These are as follows:

- The diameter of the femoral head and the acetabulum of the joints must be such that the contact pressure between the rubbing surfaces does not exceed 6.5 MPa. The desired diameter can be determined according to Equation (3).
- In the manufacturing of joints, it is necessary to provide the minimum possible clearance between the friction surfaces, that is, to manufacture parts with the highest possible accuracy.

TECHNOLOGY OF MACHINING OF THE FEMORAL HEAD OF A HIP JOINT REPLACEMENT PROSTHESIS, MADE OF GRADE 2 TITANIUM ALLOY

The obtained results indicate that pure titanium, modified by the TDN, can be successfully applied to manufacture parts of friction pairs dedicated to endoprosthesis; therefore, the development of the technology of machining especially femoral heads is an urgent task. It should be noted that up to now, such technology has not been created. The reason for this is the highly unsatisfactory machinability of titanium GRADE 2 by abrasive methods. The increased tendency to "grasp " (this process occurs as a result of a deep pulling out of the material, transferring it from one friction surface to another) leads to the instability of the machining process and unsatisfactory quality of the treated surface for practically all structural and instrumental materials.

The technological route of the GRADE 2 finishing process path should include preliminary precision machining operations aimed at obtaining product precision and operations whose purpose is to obtain surface roughness defined by Ra = 0.05 μm, which corresponds to ISO 7206-2-2005 (polishing).

The practice of manufacturing ceramic heads of endoprosthesis has shown that the free lapping (**Fig. 11**) has proved to be quite effective in obtaining the required precision of the product [**L. 21**]. Its advantage is that it does not require complex and costly equipment and may be implemented on a universal basis.

The tool (3) fixed in the clip (4) (which is supported by the hinge 5) is pressed at an angle (α) to the machined head (1) planted on the mandrel (2) mounted in the lathe chuck. A characteristic feature of the system is the low speed of the relative movement of the tool and the workpiece.

The main task of creating an abrasive tool for the processing of workpiece from GRADE 2 was the creation of abrasive composite, which avoids the accumulation and deterioration of surface quality of the surface to be processed.

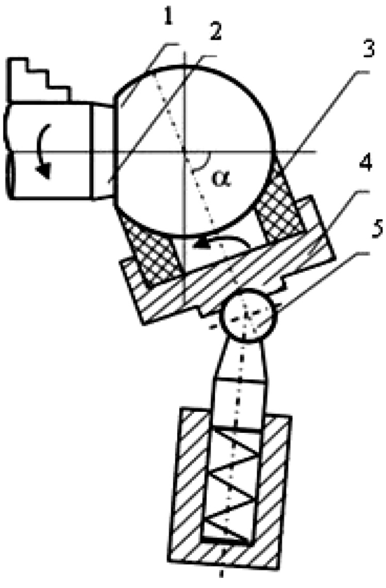

Fig. 11. Scheme of free lapping
Rys. 11. Sposób swobodnego docierania

The first part of the problem can be solved by applying a bond in the abrasive tool that holds the abrasive grains firmly.

The second part is solved by the creation of special abrasive composites. When using traditional abrasive composites, the vertices of cutting grains on the tool surface are at different heights. Therefore, only a small fraction of grains are contacted with the surface to be treated, which leads to their destruction with the separation of large fragments. As the abrasive grains are destroyed, the contact area of the bond and machined surface develops, which increases the probability of occurrence and development of "the focus of cutting."

To avoid the above-mentioned negative phenomena in the processing of titanium by possibly applying abrasive composites, their bond must be capable of reducing the modulus of elasticity when the mechanical load is increased on the abrasive grains [**L. 22**].

An epoxy acrylate resin, filled with calcium carbonate powder, has the necessary property. When applied, the group of most protruding grains, without clumping, can be immersed in a bond to a greater depth, and the number of grains forming the base of the contact increases. In this case, the uniform wear of the massif of cutting grains occurs. This is not due to macro-scaling of the most protruding grains, but due to the micro-destruction of a large number of cutting edges. With the optimum composition of the material, the clearance between the bond and surface being machined is stable and sufficient to prevent the "grasp." In addition, this property should help to increase the durability of the tool.

The tools (laps) on the basis of the developed composite (**Fig. 12**) have been tested in the processing of

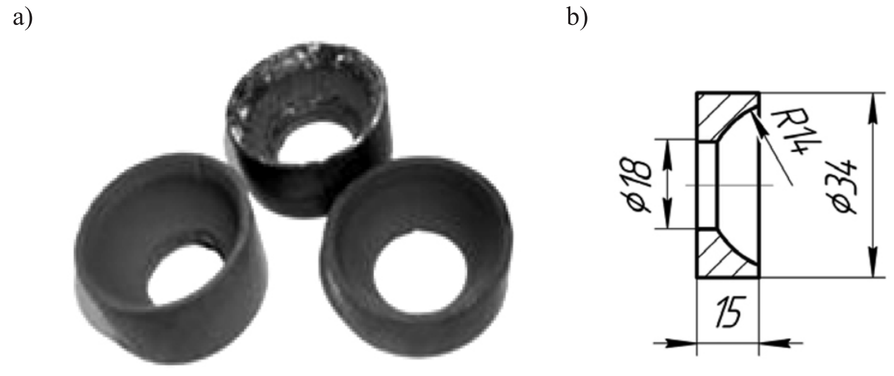

Fig. 12. Laps for processing of titanium workpiece: a) photographs of the tools, b) technical drawing of the tool
Rys. 12. Narzędzia do docierania elementów z tytanu: a) fotografie, b) rysunek techniczny

femoral heads made of GRADE 2 under the laboratory conditions at the Institute for Superhard Materials of the National Academy of Sciences of Ukraine. The tool uses synthetic diamonds AC20 at 100% concentration. The tests were carried out at the processing of Ø28 mm femoral heads. As a lubricant, "Industrial - 20" oil was used.

It has been experimentally established that the stable and safe operation of the tool based on the composite described above takes place at the rotation speed of the workpiece n = 1000 rpm.

Figure 13 shows the dependence of the intensity of material removal during machining the workpiece on the force of pressing the tools with various grains.

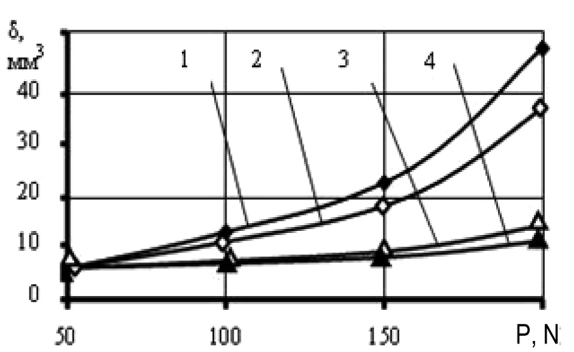

Fig. 13. Dependence of the material removal on the force of pressing for different grains of diamonds: 1) 200/160; 2) 125/100; 3) 63/50; 4) 50/40
Rys. 13. Zależność ilości usuwanego materiału od siły docisku dla różnych rozmiarów ziarna diamentu: 1) 200/160; 2) 125/100; 3) 63/50; 4) 50/40

It can be seen that the most intense wear (intensity of material removal) is provided by a tool with grain size of 200/160. Its task is to eliminate the errors of the shape of the workpiece after turning.

It has been experimentally established that the necessary condition for effective polishing of the working surface of the finished product (Ra 0.05 µm, GOST R ISO 7206-2-2005) is the roughness Ra of 0.25 µm after preliminary operations of precision machining (free lapping). This can be achieved by using smaller-grain diamonds in the tools.

The roughness of the workpiece surface was measured using an interference profilometer [**L. 23**]. In **Fig. 14,** the values of roughness of the processed surface of a femoral head are given in the sequence of applications of granularity of synthetic diamonds. The analyses performed using a scanning electron microscope (not included in the paper) did not reveal the coarse surface of the treated surface.

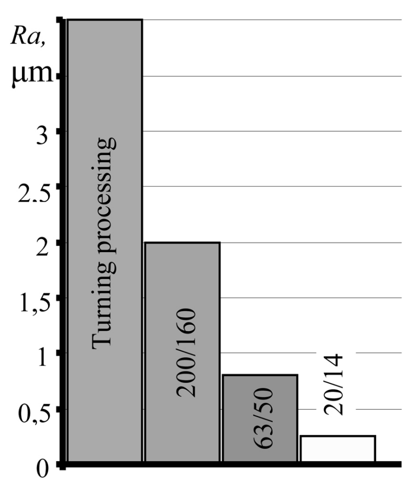

Fig. 14. Dependence of the surface roughness on the granularity of synthetic diamond; the symbols 200/160 etc. – diamond grain size in the tool
Rys. 14. Zależność chropowotaści powierzchni od granulacji sztucznego diamentu; symbole 200/160 i in. – rozmiary ziarna diamentu

A polishing paste used to polish the heads was developed at the Institute for Superhard Materials of the National Academy of Sciences of Ukraine. The paste has an intense mechanical-chemical effect on the treated surface. It is based on an active complexing agent capable of selectively extracting atoms of titanium from oxidized films on the surface; therefore, the process proceeds without the formation of defects in the deeper layers.

The mechanism of the influence of the complexing agent in the polishing paste on the intensity of the removal of allowance during the processing of titanium is associated with the peculiarities of the structure of the surface oxide film, which always exists, being self-replicating on the surface of the titanium in oxygen-containing media. Polishing waste analysis revealed that, in addition to microscopic polishing products, nanosized Ti_xO_y clusters are present. The presence of these particles is a direct consequence of the capture of the titanium atoms by the molecules of the complexing component on the boundary of the metal-paste contact. Due to the high energy of the Ti-O bond, metal titanium–oxygen clusters are removed from the surface of the metal, which are immobilized as part of multicore complex compounds.

Removing clusters of Ti_xO_y from the surface of the oxide film gives some porosity. One can assume that the intensity of the removal of allowance, as well as the roughness of the treated surface of titanium at the nanoscale level, will be related to the distribution of the surface pores of the equilibrium oxide film in dimension.

The appropriate distribution for GRADE 2 samples that were polished using paste with different complexing content was obtained by the Barrett-Joyner-Halenda (BJH) method. The results shown in **Fig. 15** correspond to small (1), optimal (2), and high content of the complexing agent in the paste. The total porosity of the equilibrium oxide film for these three products corresponds to 1: 1.8: 1.6.

Thus, the use of a polishing paste with an optimal content of the complexing agent made it possible to form the GRADE 2 polished surface with the highest efficiency. In this case, the roughness of the formed surface corresponds to the minimum size of the heterogeneities of the oxide film.

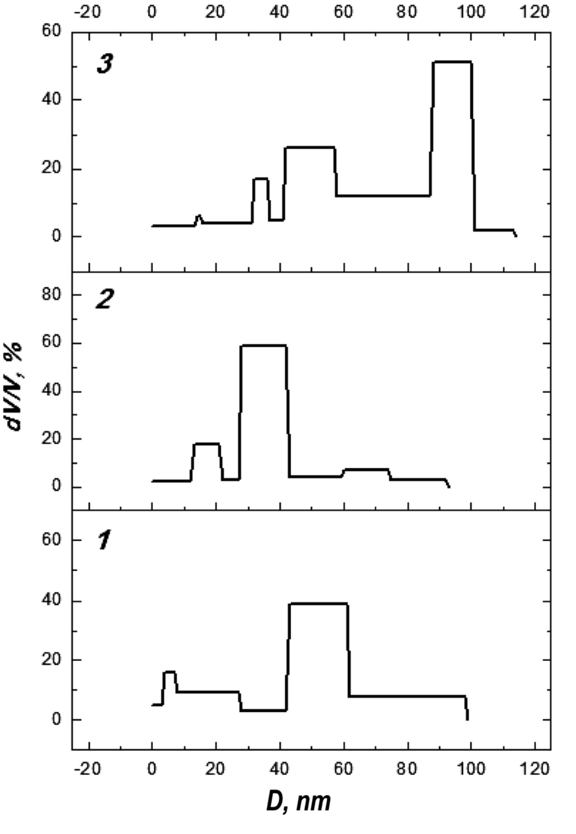

Fig. 15. Distribution of the surface pores with small (1), optimal (2), and high content (3) of the complexing agent in the paste; *dVV*, % - is percentage of pores, and *D*, nm - is the size of the investigated area

Rys. 15. Rozkład porów w powierzchni dla małego (1), optymalnego (2) i wysokiego (3) stężenia środka kompleksującego w paście polerskiej; *dVV*, % – procentowa porowatość, *D*, nm – rozmiar analizowanej powierzchni

The results of measurements of the surface roughness parameters after polishing are given in **Tab. 3**, and **Fig. 16** shows a photograph of the balls manufactured according to the developed technology.

Table 3. Surface roughness parameters after polishing; the definitions of the parameters can be found in ISO 25178-2:2012

Tabela 3. Chropowatość powierzchni po polerowaniu; parametry chropowatości zostały zdefiniowane w normie ISO 25178-2:2012

Sq [µm]	*Ssk*	*Sku*	*Sp* [µm]	*Sv* [µm]	*Sz* [µm]	*Sa* [µm]	*Rz* [µm]	*Ra* [µm]
0.0725	–1.66	9.85	0.243	0.62	0.863	0.524	0.2	0.04

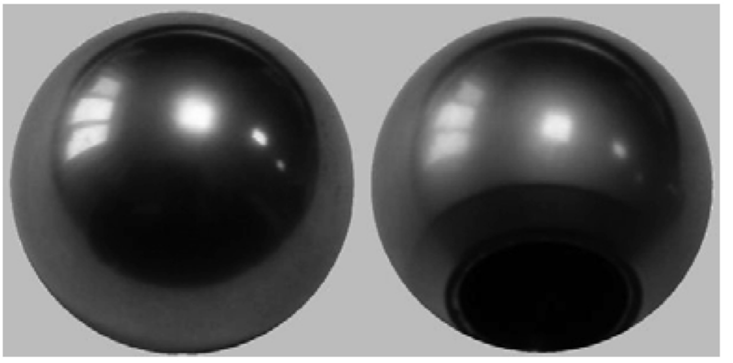

Fig. 16. Femoral heads of endoprosthesis (GRADE 2)
Rys. 16. Główki endoprotez (GRADE 2)

It can be seen that the product also meets the requirements of the GOST R ISO 7206-2-2005 standard with respect to the surface roughness.

SUMMARY AND CONLUSIONS

The paper presents the first part of the work performed within the Polish-Ukrainian cooperation. The following conclusions can be drawn at this stage:

1. Anti-friction properties of the friction pair "Nitrided GRADE 2/PE-UHMW" substantially exceed the traditional "CoCrMo/PE-UHMW" pair, which is important for endoprosthetic practice. Friction in a pair of "Nitrided GRADE 2/PE-UHMW" is lower by 25%, and wear is lower by 60%. After passing 200 km of sliding distance, which corresponds to ~23 million cycles of load, the wear of the titanium component was not detected. This suggests that the modification of the surface of the titanium alloy GRADE 2 makes it possible to compete even with the ceramic components in the friction pair containing PE-UHMW.
2. The new working fluid based on chondroitin sulphate can be successfully used in studies of tribological characteristics of friction pairs "CoCrMo/PE-UHMW" and "Nitrided GRADE 2/PE-UHMW," using bench friction machines and simulator stands.
3. To achieve the smallest wear in the "Nitrided GRADE 2/PE-UHMW" endoprosthesis, the diameter of the head and the acetabulum must be such that the contact pressure between the rubbing surfaces does not exceed 6.5 MPa. It is also necessary to provide the minimum possible clearance between the friction surfaces, that is, to manufacture the head and acetabulum with the highest possible accuracy.
4. Artificial composites based on modified epoxy resins and synthetic diamonds, representing three-level adaptive systems, allow the processing of technically pure titanium by free lapping without shrinking and scratching the treated surface, providing a roughness of *Ra* 0.25 μm and an accuracy up to 0.008 μm (in accordance with GOST R ISO 7206-2-2005).
5. Application of the polishing paste developed at the Institute for Superhard Materials of the National Academy of Sciences of Ukraine allows one to obtain the roughness of the working surface of the GRADE 2 head of $Ra \leq 0.05$ μm, which is required by GOST R ISO 7206-2-2005.

The second part of the work is focused on verification testing of the developed biomaterial using a hip joint simulator. In Poland, there are two research institutes most active in tribological testing of hip joint endoprosthesis, having appropriate test devices: Metal Forming Institute in Poznan and Institute for Sustainable Technologies – National Research Institute in Radom. The hip joint simulators from these two organisations are described in, e.g., [**L. 24**], as well as in [**L. 25, 26**], respectively. Similar research is also performed in Bialystok University of Technology [**L. 27**].

REFERENCES

1. Miletic K.G., Taylor T.N., Martin E.T., Vaidya R., Kaye K.S.: Readmissions after diagnosis of surgical site infection following knee and hip arthroplasty. Infect Control Hosp Epidemiol., Vol. 35, 2014, pp. 152–157.
2. Pinchuk L.C., Nikolaev V.I., Tsvetkova E.A., Goldade V.A.: Tribology and biophysics of artificial joints. Elsevier. London 2006.
3. Filippenko V.A., Tankut A.V.: Evolution of joint prosthetics problem. International Medical Journal, No. 1, 2009, pp. 70–74.

4. Charnley J.: Arthroplasty of the hip. A new operation. Lancet, No II, 1961, pp. 54–56.
5. Nadeev A., Ivannikov S.V.: Endoprostheses of the hip joint in Russia: the construction philosophy, implant review, rational choice. Knowledge Lab, 2015 (electronic source).
6. Kurtz S.M.: Ultra high molecular weight polyethylene in total joint replacement and medical devices. In: UHMWPE Biomaterials Handbook. Academic Press. Burlington, 2009.
7. Goodman S. et al.: Tissue in growth and differentiation in the bone harvest chamber in the presence of CoCr alloy and high density polyethylene particles. J. Bone Joint Surg, 77A, 1995, pp. 1025–1035.
8. Mc Kee G.K.: Artificial hip joint. J. Bone Joint Surg., No. 33-b, 1951, p. 465.
9. Pollyah M.V.: Endoprosthetics of the hip joint. http://rheumatology.com.ua/blog/articles/735.
10. Chechulin B.V., Ushkov S.S., Razuvaev I.N., Goldfain V.N.: Titanium alloys in mechanical engineering. Mashinostroenie, 1977.
11. Filippenkova V.A., Tankut A.: Evolution of the problem of endoprosthetics of joints. International Medical Journal, No. 1, 2009, pp. 70–74.
12. The study on the creation of a new method of arthroplasty of the hip joint. Proc. conf. specialists in osteoarticular tuberculosis of Moscow, 1956, pp. 16–52.
13. Pohrelyuk I.M., Sheykin S.E., Dub S.M., Mamalis A.G., Rostotskii I.Yu., Tkachuk O.V., Lavrys S.M.: Increasing of functionality of c.p. titanium/UHMWPE tribo-pairs by thermodiffusion nitriding of titanium component. Biotribology, vol. 7, 2016, pp. 38–45.
14. Sheykin, S.Ye., Pohreliuk, I.M., Iefrosinin, D.V., Rostotskiy I.Yu., Sergach, D.A.: The use of titanium in the friction units of artificial joints. Sci. innov., 11(3), 2015.
15. Fedirko V.M., Pokerglyuk I.M.: Nitration of titanium and its alloys. Scientific opinion, 1995.
16. Ermakov F.: Tribological physics of liquid crystals. IMMS of the National Academy of Sciences of Belarus, Gomel, 2008.
17. Krugelsky I.V., Dobychin M.N., Kombalov V.C.: Basics of calculations for friction and wear. Mechanical Engineering, 1977.
18. Chernyakova Yu.M., Pinchuk L.S.: Synovial joint as a clever friction unit. Friction and wear, vol. 28, No. 4, 2007, pp. 410–417.
19. Ermakov S.F., Beletsky A.V.: Tribological principles of blood serum preparations as a liquid crystalline medium for therapeutic correction of synovial joints. Friction and wear, vol. 32, No. 1, 2011, pp. 65–71.
20. Grushko A.V., Sheykin S.E., Rostotskiy I.Y.: Contact pressure in hip endoprosthetic swivel joints. J. Frict. Wear, 33, 2012, pp. 124–129.
21. Novikov N.V., Rosenberg O.A., Gavlik J.: Endoprostheses of the human joints: Materials and technologies. ISM them. VN Bakul NAS of Ukraine, Kyiv, 2011.
22. Paschenko Ye.A., Sheykin S.Ye., Iefrosinin D.V.: Tools for precision diamond machining of spherical heads of endoprostheses from pure titanium. Journal of Superhard Materials, vol. 35-3, 2013, pp. 175–182.
23. Ignatovich S.R., Zakiev I.M.: Surface quality control using an interference profilometer. Inventor and innovator, No 6, 2007, pp. 8–11.
24. Wisniewski T., Mroz A., Magda J., Wielowiejska-Giertuga A.: Stand for tribological testing of hip joint endoprostheses. Tribologia, No. 6, 2016, pp. 167–177.
25. Niemczewska-Wojcik M., Piekoszewski W.: The surface topography of a metallic femoral head and its influence on the wear mechanism of a polymeric acetabulum. ACME, vol. 17, 2017, pp. 307–317.
26. Niemczewska-Wojcik M.: Wear mechansisms and surface topography of artificial hip joint components at the subsequent stages of tribological tests. Measurement, vol. 107, 2017, pp. 89–98.
27. Gradzka-Dahlke M.: Exploatative properties of a new cup of hip joint endoprosthese. Scientific Journals of Maritime University of Szczecin, 19(91), 2009, pp. 47–50.