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THE EFFECT OF AN ACCELERATED AGEING PROCESS ON TRIBOLOGICAL WEAR OF POLIMERIC ELEMENTS IN JOINT ENDOPROSTHESES

WPLYW PROCESU PRZYSPIESZONEGO STARZENIA NA ZUŻYCIE TRIBOLOGICZNE POLIMEROWYCH KOMPONENTÓW ENDOPROTEZ

Key words:

accelerated ageing, UHMWPE, friction and wear tests, wear debris

Słowa kluczowe:

przyspieszone starzenie, UHMWPE, badania tarciovo-zużyciowe, produkty zużycia.

Abstract

The polymeric components of endoprostheses in in vivo conditions are subjected to the oxidation process (ageing). The paper presents the results of

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friction and wear testing performed with use of a knee-joint simulator. The polymeric parts were machined from UHMWPE and UHMWPE modified with the addition of vitamin E. The calorimetric analysis and tribological testing results confirm the negative effect of accelerated ageing in oxygen atmosphere on the tribological properties of UHMWPE as well as the antioxidative properties of vitamin E

INTRODUCTION

The knee joint is one of the most complex bio bearings that is sensitive and vulnerable to damage [L. 1, 2, 3]. One of the most effective treatments for injuries and deformities that lead to degradation of the articular surface and changes in the subchondral layer is knee joint arthroplasty [L. 4].

Currently, there are several endoprosthesis construction solutions. They differ in friction surface geometry, the materials used, and the degree of taking over the functions of the damaged joint. Femoral components and tibial endoprostheses are produced mainly from the Co28Cr6Mo alloy, due to its greater resistance to tribological wear in comparison to other alloys, e.g., Ti6Al4V. The movable element (inlay) is made of ultrahigh molecular weight polyethylene (UHMWPE). The use of inlays made of UHMWPE reduce the rigidity of the system and provide better shock vibration absorption due to the properties of this material. The use of metal-polyethylene material combinations provides relatively low frictional resistance [L. 5].

The most essential prerequisite for the preparation of implant materials is sterilization. In the case of polymeric materials, radiation sterilization is considered a highly effective method. Gamma radiation results in a change in UHMWPE cross-linking, which affects the molecular weight, the degree of crystallinity, the modulus of elasticity, and other physical and chemical properties [L. 6, 7]. A side effect of the sterilization process is the generation of free radicals. The presence of free radicals in the UHMWPE structure causes connections between adjacent chains to form due to the reaction with oxygen. This leads to the deterioration of the mechanical properties, reducing the density and resistance to tribological wear [L. 8].

Taking into account the problems defined over the years associated with the use of UHMWPE for endoprosthesis elements, various treatments in the manufacturing process of the material have been proposed. They include three-step annealing [L. 9], plastic deformation in the solid state [L. 10], or vitamin E doping [L. 11].

The available data indicate that vitamin E modified polyethylene has a higher resistance to oxidation. This is confirmed by the structural and strength research results [L. 12]. Despite the fact that the material has been used for endoprostheses for several years, the tribological properties of the material are

still not sufficiently known. Admittedly, the first clinical reports already available seem to confirm the beneficial effects of vitamin E; however, they refer to short-term clinical trials [L. 13].

Considering the above-described problems, the article presents an analysis of the effect of ageing UHMWPE and UHMWPE modified with vitamin E on the resistance to tribological wear. The aim of the study was to determine the effect of ageing on the tribological properties of UHMWPE. The conducted comparative tests made it possible to confirm the thesis of the validity of the idea of UHMWPE modification with vitamin E, which aims to reduce UHMWPE susceptibility to oxidation.

SUBJECT AND RESEARCH METHODOLOGY

The investigated materials were inlays produced by machining from ultrahigh molecular weight polyethylene (UHMWPE) and UHMWPE modified with the addition of vitamin E (hereinafter UHMWPE-vit. E) rods, which were provided by a European manufacturer of endoprostheses. To study the thermal properties, both sterilized and unsterilized inlays were applied, whereas for tribological testing purposes, only sterilized ones were used.

The thermal properties, such as crystallization temperature T_C , melting point determined in the first and the second heating cycle, T_{M1} and T_{M2} , and melting enthalpy ΔH_m as well as the degree of crystallinity X_C , were determined by differential scanning calorimetry (DSC). A Netzsch DSC 204 F1 Phoenix camera was employed to execute the tests, and measurements were carried out in an inert atmosphere (nitrogen) in accordance with the following procedure: heating to 230°C at a heating rate of 10°C/min, isothermal annealing at 230°C for 5 min and cooling to a temperature of 20°C at 10°C/min. Samples weighing $5 \text{ mg} \pm 0.1 \text{ mg}$ were used for the investigations. The procedure was repeated twice in order to eliminate the effect of processing on the phase transformation temperature. The degree of crystallinity was determined according to the following equation [L. 14]:

$$X_c = \frac{\Delta H_m}{\Delta H_m^0} \cdot 100\% \quad (1)$$

where ΔH_m is the melting enthalpy of the sample, and ΔH_m^0 the UHMWPE melting enthalpy value amounting to 291 J/g [L. 15].

Friction and wear tests were carried out using a simulator to study the tribological properties of knee joint endoprostheses, SBT-02.1 [L. 16]. The construction of the simulator is based on the anatomy of the knee, taking into

account the characteristics of the variable load during human gait. The most important parameters of the simulation during the test are the number of cycles (1 000 000), the frequency (1 Hz), extension-flexion angle range (from -20° to $+20^\circ$), and the instantaneous maximum normal load (1 kN). The tests were carried out at the temperature of $23 \pm 1^\circ\text{C}$ in a medium of distilled water. The subject of friction and wear research consisted of 12 sets of "inlay-femoral endoprosthesis component". Six inlays are made of UHMWPE, and another six were made of UHMWPE-vitamin E. Half of each batch was subjected to an accelerated ageing process, which was conducted in accordance with ASTM F2000-02 [L. 17]. The most important process parameters are oxygen atmosphere, pressure 503 kPa, temperature 70°C , and a time of 336 ± 1 h. The femoral components (diameter 40 mm) are made of Co28Cr6Mo alloy.

Immediately before and after the tribological tests, the samples were washed, dried, and weighed according to the protocol described in ISO 14242-2 [L. 18]. Mass measurements were performed using a laboratory scale EX 2225DM (Ohaus Corp., USA) with a resolution of 0.00001 g. In order to minimize the effect of lubricant sorption during the tribological tests and its impact on the defining the relevant wear of the tested materials, the samples were immersed in a lubricating fluid for five days before testing.

The results of the friction and wear tests consisted in comparing the course of changes in the friction coefficient value as a function of sliding distance (number of cycles), and the wear rates. The measuring system used in the simulator allows one to determine the coefficient of friction based on the recorded friction torque values. The wear rate value was determined based on the wear volume quotient by multiplying the average value of the loading force and sliding distance.

Before proceeding to the tribological tests, analysis of the Hertz maximum stress and maximum shear stress distribution in the initial testing phase at the moment of loading the tribological system with the maximum shear force was conducted. The following equation was used for the calculations [L. 19]:

$$b = \sqrt{\frac{2F(1-\nu_w^2)/E_w + (1-\nu_u^2)/E_u}{\pi(1/d)}} \quad (2)$$

where b – contact area half the width of the inlay, F – loading force, E_w and E_u – Young's modulus respectively for the inlay material and the femoral component, and ν_w ν_u – Poisson's ratio respectively for the inlay material and the femoral component, l – width of the contact surface of the femoral component, d – diameter of the femoral component,

$$p_{\max} = \frac{2F}{\pi bl} \quad (3)$$

where p_{\max} – the maximum shear stress,

$$\sigma_x = -2\nu p_{\max} \left[\sqrt{\left(1 + \frac{z^2}{b^2}\right)} - \left|\frac{z}{b}\right| \right] \quad (4)$$

where σ_x – principal stresses in the x direction, z – distance from the surface (depth);

$$\sigma_y = -p_{\max} \left[\frac{1 + 2\frac{z^2}{b^2}}{\sqrt{1 + \frac{z^2}{b^2}}} - 2\left|\frac{z}{b}\right| \right] \quad (5)$$

where σ_y – principal stress in the y direction;

$$\sigma_z = \frac{-p_{\max}}{\sqrt{\left(1 + \frac{z^2}{b^2}\right)}} \quad (6)$$

where σ_z – the principal stress in the z direction;

$$\tau_{xz} = \frac{\sigma_x - \sigma_z}{2} \quad (7)$$

where τ_{xz} – shear stress xz;

$$\tau_{yz} = \frac{\sigma_y - \sigma_z}{2} \quad (8)$$

where τ_{yz} – shear stress yz;

Based on published data, the Young's modulus adopted for the calculations for the Co28Cr6Mo UHMWPE was 240,000 MPa and 658 MPa, respectively, and the Poisson's ratio adopted for the calculations was 0.300 and 0.355, respectively [L. 20, 21].

Analysis of the wear mechanism of the tested materials was carried out on the basis of SEM observation of the morphology of the friction surface using a scanning electron microscope Inspect S (FEI).

Measurements of surface roughness Ra in the direction perpendicular to the direction of signs of wear were performed using a Hommel profilometer ETAMIC T8000RC (Jenoptik AG). The measurements were conducted on a section of 4.8 mm. Determining the average roughness consisted in carrying out ten measurements for the first friction pair representing the given tribosystem. The subsequent pair was the only control (3 measurements) used to check whether the value of the Ra parameter is within the range defined for the first pair.

Analysis of the size distribution of the wear products was performed using a Mastersizer 3000 device (Malvern, UK). During the analysis, standard sensitivity was used, and the time to break up agglomerates was 10 s.

EXPERIMENTAL RESULTS

Table 1 shows the thermal property values of the samples determined by DSC. Particular attention should be paid to the change in the degree of crystallinity caused by sterilization. This process resulted in a significant reduction in this property in the case of the UHMWPE samples, which appreciably affected their structure. While it is difficult to talk about the degradation of the material in spite of comparable X_c and T_M values, the sterilization process significantly influenced the structure of the samples. A significant reduction in the degree of crystallinity can lead to a reduction in the hardness and a deterioration of the strength properties of polymeric materials. Regarding the UHMWPE-vit. E samples, a difference between the X_c value is also noticeable; nevertheless, it is much more limited. Both samples modified with vitamin E (sterilized and unsterilized) were characterized by a lower degree of crystallinity compared to the unmodified sample.

Table 1. Thermal properties determined by DSC
Tabela 1. Właściwości termiczne wyznaczone metodą DSC

Material		UHMWPE	UHMWPE after sterilization	UHMWPE-vit.E	UHMWPE-vit.E after sterilization
T_C		116.2	112.5	115.8	115.9
T_{M1}	[°C]	138.9	144.0	143.8	143.9
T_{M2}		134.9	136.8	136.0	135.7
ΔH_{M1}	[J/g]	183.5	150.2	166.7	155.3
ΔH_{M2}		164.6	160.3	156.2	156.3
X_{C1}	[%]	63.1	51.6	57.3	53.4
X_{C2}		56.6	55.1	53.7	53.7

From the analysis of the available literature data, it appears that the properties that determine the fatigue strength of UHMWPE, and thus the resistance to tribological wear, are the degree of crystallinity and hardness [L. 22, 23]. Calorimetric investigations, based on which the degree of crystallinity of the tested materials was calculated, showed that in the UHMWPE structure, as a result of the sterilization process, there is a significant decrease in the degree of crystallinity. To determine the exact structural changes, isothermal and non-isothermal crystallization kinetics testing by the Arvami method should be carried out [L. 24]. This analysis allows one to present a detailed description of the observed changes. Conducting complementary testing by wide angle X-ray diffraction (WAXS), enabling the assessment of structural and morphological changes occurring as a result of inlay modification and sterilization, as well as spectroscopic investigations (FT-IR) in order to preclude degradation of the polymer material caused by the sterilization process and describe the chemical changes induced by modification, should also be considered.

In the initial phase of tribological testing, the contact of the friction surfaces of the femoral component and the polymeric inlay was linear. Moreover, the stress values caused by the loading force and as a consequence of the contact geometry reached maximum values. For all the tested friction pairs, the maximum Hertz stress value was 19.9 MPa, and thus did not exceed the maximum permissible value (20 MPa for UHMWPE). For both the femoral component and the polymeric inlay, the maximum shear stress, amounting to 6 MPa, occurred at a distance of 0.84 mm from the contact surface.

By analysing the mean friction coefficient values, it can be said that the UHMWPE-Co28Cr6Mo friction couple is characterized by the lowest friction resistance among all the tested material combinations (0.053). The ageing process resulted in an increase in the friction coefficient to 0.069. In the case of the friction pair with the participation of UHMWPE-vitamin E, after the sterilization process, the applied ageing process resulted in a slight increase in the friction coefficient from 0.064 to 0.068. The median values for each friction pair against the spectrum of values (from minimum to maximum) are shown in **Fig. 1a**.

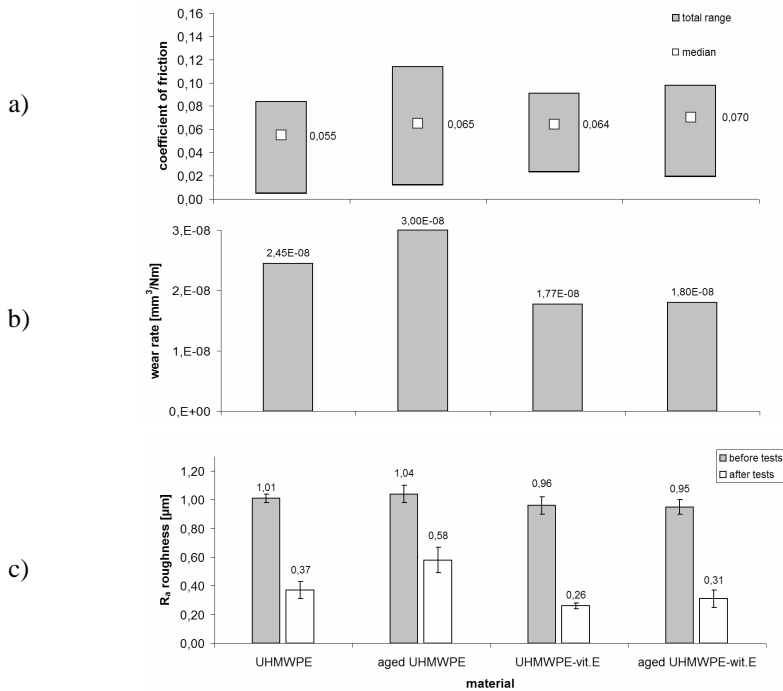


Fig. 1. Median coefficient of friction (a), wear rate (b), and R_a roughness (c) in relation to inlay material

Rys. 1. Mediana współczynnika tarcia (a), wskaźnik zużycia (b) oraz chropowatość R_a (c) w zależności od materiału wkładki

As a result of ageing, the median coefficient of friction for the UHMWPE-Co28Cr6Mo pair increased from 0.055 to 0.065 (an increase of approximately 18%). For comparison, the median friction coefficient for the UHMWPE-vit. E – Co28Cr6Mo pair due to the ageing process increased from 0.064 to 0.068, which is approximately 6% of the value of this parameter.

The average value of the loading force during all the tests was about 920 N. The apparent density of UHMWPE and UHMWPE-vit. E amounted to 9.38 and 9.40 g/cm³, respectively. Fig. 1b shows the comparison of the mean wear rate values. Based on the results, it can be concluded that the increased coefficient of friction for the tested friction pairs as a result of ageing UHMWPE and UHMWPE-vit. E is reflected in an increase in susceptibility of these materials to the amount of generated wear products. The wear rate value for the UHMWPE modified with vitamin E increased slightly, while UHMWPE observed more than a tenfold increase in the value of this parameter.

The initial roughness of the friction surfaces of the femoral component and polymer inlays fulfilled the assumptions of standard ASTM F732 [L. 22], according to which the maximum recommended R_a parameter values for metal

and polymer endoprostheses are 0.05 and 1.60 μm , respectively. As a result of these tests, the roughness of the friction surfaces of the metal components increased. In the case of the polymer samples, an opposite trend was observed – the Ra parameter was characterized by lower values after the tribological tests compared to the state before their commencement. **Fig. 1c** shows a comparison of Ra roughness of the polymer samples before and after ageing.

The decrease in the Ra values as a result of smoothing the surfaces during friction was more pronounced for the UHMWPE-vit. E inlays in comparison to the UHMWPE inlays. The roughness of the components that were aged before testing was greater than those that were not aged.

Fig. 2 shows SEM images of the inlay surfaces morphology. Before commencing tribological testing, traces of finishing treatment were observed on the friction surfaces (**Fig. 2a** and **2b**).

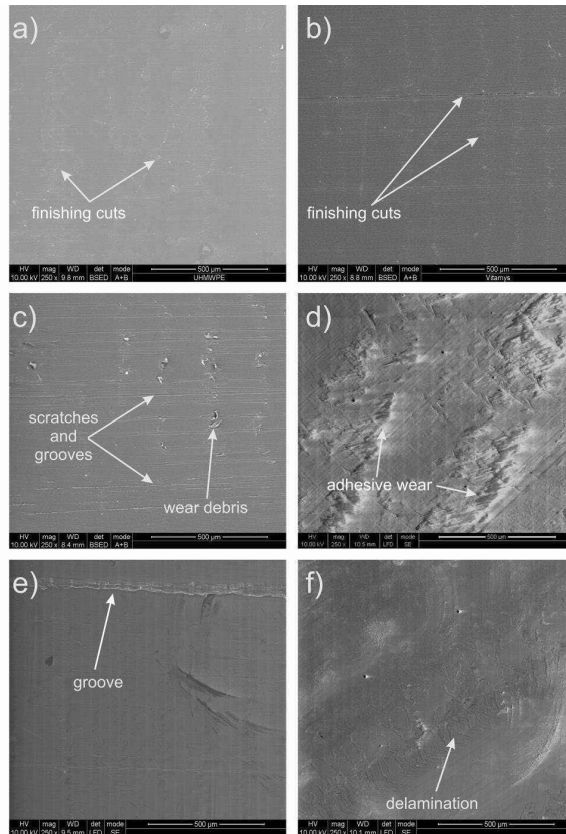


Fig. 2. Morphology of surfaces after tribological testing: a) UHMWPE, b) aged UHMWPE, c) UHMWPE-vit. E, d) aged UHMWPE-vit. E

Rys. 2. Morfologia powierzchni trących po zakończeniu testów tribologicznych: a) UHMWPE, b) UHMWPE starzony, c) UHMWPE-vit. E, d) UHMWPE-vit. E starzony

The visible traces of scratching and grooving are the result of the presence of hard metal wear products in the friction couple. Comparing the image of the surface morphology of the UHMWPE (**Fig. 2c**) and UHMWPE-vit. E (**Fig. 2e**) inlays, it can be concluded that there is less friction and wear for the second material.

The process of the accelerated ageing of UHMWPE contributed to its more intensive wear. **Fig. 2d** reveals clear signs of adhesive wear. The SEM images of the UHMWPE-vit. E inlays confirm that this material has a higher resistance to tribological wear (**Fig. 2d**). On the friction surfaces, only a few signs of wear in the form of scratches and delamination are visible. The ageing process of UHMWPE-vit. E did not contribute to such a clear deterioration in resistance to tribological wear as it did in the case of polyethylene without the addition of vitamin E.

Figure 3 shows a comparison of the size distribution of the tribological wear products of the tested elements.

In the case of UHMWPE and aged UHMWPE, the dimensions of the wear products are contained within the range from 0.4 to 200 μm (**Fig. 3a**). For both materials, the size distribution had a bimodal character. The ageing process contributed to the generation of more wear products of larger sizes due to friction. The largest share of particles was characterized by particles having a diameter from 20 to 30 μm . The $d_v(90)$ percentile value was about 50 μm .

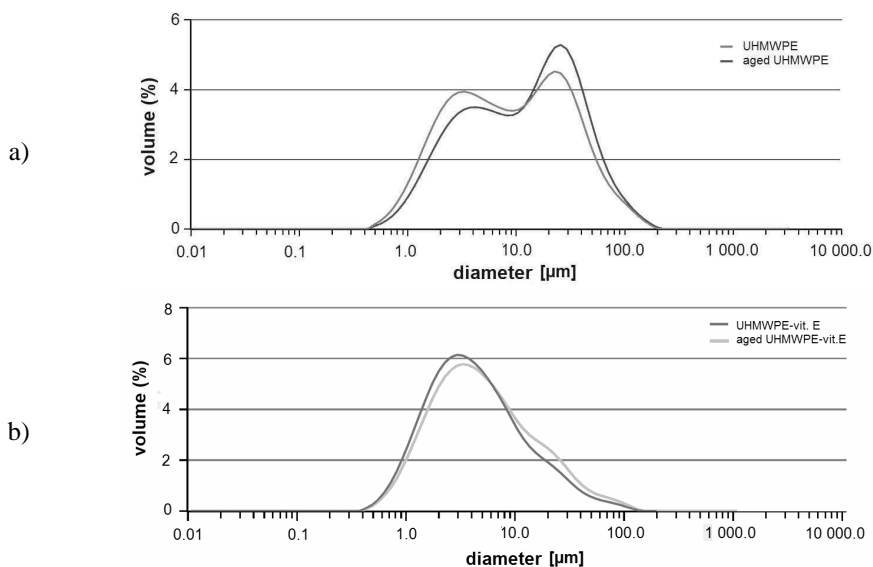


Fig. 3. Wear debris size distribution of: a) UHMWPE, b) UHMWPE-vit. E

Rys. 3. Rozkład wielkości produktów zużycia przed i po procesie starzenia: a) UHMWPE, b) UHMWPE-wit. E

The modification of UHMWPE with vitamin E helped to change the nature of the modal distribution, and most of the generated wear products were characterized by smaller dimensions (**Fig. 3b**). The largest share of wear was characterized by products with a diameter of 3 to 4 μm . The d_v (90) percentile value was approximately 25 μm .

The ageing process of both tested materials contributed to the generation of wear debris of larger sizes. Therefore, the peaks shift towards the higher values of the distribution. It should be noted, however, that this shift was more pronounced for UHMWPE compared to UHMWPE-vit. E.

CONCLUSIONS

The tests conducted by the authors of were comparative in nature, and the purpose of the study was to determine the effect of accelerated ageing on the resistance to the tribological wear of UHMWPE and UHMWPE modified with vitamin E. The results obtained allow us to draw the following conclusions:

1. As a result of accelerated ageing in an oxygen atmosphere, the wear resistance of the polymeric materials used in the components of knee-joint endoprostheses deteriorates.

2. The calorimetric and tribological tests confirmed the validity of modifying UHMWPE with the addition of vitamin E.

3. The antioxidant properties of vitamin E helped to limit the phenomenon of reducing UHMWPE crystallinity resulting from accelerated ageing, as reflected in a reduction of the tribological parameters, such as friction, wear rate, or the dimensions of wear debris generated during friction.

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Streszczenie

Polimerowe komponenty endoprotez w warunkach in vivo ulegają procesowi starzenia (utleniania). W artykule przedstawiono wyniki badań tarciowo-zużyciowych endoprotez stawu kolanowego. Komponenty polimerowe wykonano z UHMWPE i UHMWPE modyfikowanego witaminą E. Wyniki badań kalotometrycznych i tribologicznych potwierdziły negatywny wpływ procesu przyspieszonego starzenia w atmosferze tlenu na właściwości strukturalne i tribologiczne UHMWPE oraz antyoksydacyjne właściwości witaminy E.

