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THE EVALUATION BY MEANS OF MICROINDENTATION AND SCLEROMETRY OF CHANGES IN PROPERTIES AFTER ELECTRON BEAM IRRADIATION OF POLYETHYLENE GUR 1050

DIAGNOZOWANIE METODĄ MIKROINDENTACJI I SKLEROMETRII ZMIAN WŁAŚCIWOŚCI PO NAPROMIENIOWANIU STRUMIENIEM ELEKTRONÓW POLIETYLENU GUR 1050

Key words:

radiation modification; operational load; polymer deformation; abrasive wear micromechanism; resistance to wear ratio; hardness; Young's modulus

Słowa kluczowe:

modyfikacja radiacyjna obciążenie eksploatacyjne; odkształcenie polimeru; mikromechanizm zużycia ściernego; wskaźnik odporności na zużycie; twardość; moduł Younga

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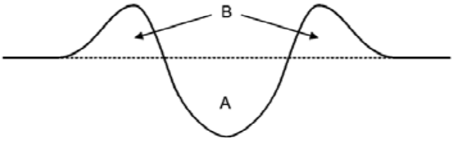
Summary

With the aim to increase the resistance of GUR 1050 polyethylene to plastic deformation and abrasive wear, modification through radiation was applied. Compression tests of up to 300 MPa showed an increase in resistance to permanent deformation. This has been confirmed with microindentation and sclerometry tests. Scratch test indicated a lower tendency of the carved material to chipping than to uplift in all operational deformations. The domination of the micromechanism of the formation of ridges allows the material to retain a small decrease in resistance to wear. The parameters of microtests may be used for the purpose of the evaluation and selection of the polymers used in friction joints in medicine and technology.

INTRODUCTION AND EXPERIMENTAL PROCEDURE

The operational durability of polymer and metal kinematic systems (e.g. endoprostheses for human joints) depends mainly on polyethylene resistance to plastic deformation and abrasive wear **[L. 1–8]**. Recently, a GUR 1050 polymer has been introduced for the production of medical implants. It is therefore justified to examine the susceptibility of the material to deformation as well as the possibility to improve its properties by means of its irradiation with an electron beam. The GUR 1050 polymer, which has been marked as BZ_{50} in its initial (base) state, was subjected to analysis. Tests were conducted on deformed samples of initial material (BZ_{50}) and on the material irradiated with an electron beam before radiation (NO_{50k}). The index 50 corresponds to the tested grade of polymer, letters N and O indicate irradiation and deformation, and k = 1-4 means the multiplication factor of irradiation with a dose of 26 kGy. Modification through radiation was performed using a linear accelerator Elektronika 10/10 (energy of electrons 10 MeV; beam power 10 kW). The samples were compressed with the rate of 5 mm/min at room temperature. Total compressions of Z_{tot} of 40, 50, 60, 70, and 80% were applied (three samples were prepared for each compression intended). The compression Z_{tot} is defined by $Z_{tot} = 100 \cdot \Delta h/h_0$, where h_0 is the initial height of the sample, h_1 is the height of sample under maximum load, and $\Delta h = h_1 - h_0$ is the height reduction due to compression. After reaching the assumed compression, the sample was immediately unloaded. The load P_{tot}, corresponding to the applied compression Z_{tot}, was recorded and the nominal stress, $\sigma_{tot} = P_{tot}/A_0$ (A_0 is the initial cross-section area of the sample), was calculated. True plastic strain is defined as $e_f = \ln(h_0/h_f)$, where h_f means the final height of the samples after elastic recovery. The recovered elastic deformation component Z_{el} was determined as a difference between Z_{tot} and Z_{ef}, where $Z_{ef} = (h_0 - h_f)/h_0 \cdot 100\%$.

Micromechanical and sclerometric properties of the polymer were determined with the use of a Micron-Gamma machine equipped with an additional self-levelling table. This solution ensures that the surface of the sample and the base of the measuring table are parallel to each other, which significantly increases the accuracy of the measurements. In microindentation tests, a Berkovich penetrator was used, load 1N, and at a load time under maximum pressure of 15s. To determine the hardness *H* and the elasticity modulus *E*, the Oliver-Phare method was applied. The algorithm approximating the curve of unloading covered an analysis 70% of its range for the elastic-plastic contact. The measurement results were averaged for 7 indentations. In sclerometric tests, the arrowhead of the triangular blade in Berkovich indenter was set motion-wise. During the scratch test, a nominal power of 2.5 N and a scratch speed of 90 μm/s were applied. The measurement of the indented surface A and of the plastic uplift was performed with the use of Taylor Hobson profilographometer equipped with TalyMap Universal software. This allowed the authors to determine the abrasive wear micromechanism β as well as the wear resistance ratio W_β (1) and (2) **(Fig. 1)**.



$$\beta = \frac{1}{n}\sum_{i=1}^{n}\frac{A_i - B_i}{A_i} \quad [-] \qquad (1)$$

$$W_\beta = \frac{1}{\frac{1}{n}\sum_{i=1}^{n}(\beta_i A_i)} \quad [mm-2] \qquad (2)$$

Fig. 1. A cross-section of a scratch resulting from a scratch test
Rys. 1. Schemat przekroju poprzecznego rysy powstałej podczas scratch testu

RESULTS AND DISCUSSION

The results of the compression test were approximated with a regression equation (R ≥ 0.97) expressing the stress σ_{tot} in deformation function Z_{tot}, Z_{el}, e_f, and then transforming these relations to the following (3) – (5) (**Figs. 2–4**):

$$Z_{tot} = \frac{1}{0.06}\ln\left[\frac{1}{w}\left(\sigma_{tot} - \frac{\pi R_e}{2}\right)\right] \quad [\%] \qquad (3)$$

$$e_f = \frac{1}{n}\ln\left(\frac{2}{\pi R_e}\sigma_{tot}\right) \quad [-] \qquad (4)$$

$$Z_{el} = \frac{1}{0.6}\ln\left[\frac{1}{w_{el}}(\sigma_{tot} - 2.5R_e)\right] \quad [\%] \qquad (5)$$

where:

- R_e (=21MPa) – the plasticity limit of the GUR 1050 polyethylene,
- w (=w_0+w_k) – the increase in the intensity of the stress with total unitary deformation of the initial material (w_0 = = 1.78 MPa / % for BZO_{50}) and the irradiated material ($w_k = 0.16\sqrt{k}$ for $NO_{50.k}$),
- k (=1-4) – multiplication factor of the electron beam irradiation dose (d = 26kGy),
- n (= n_0 + n_k) – increase in the ratio of the resistance put up by the material with the increase of plastic deformation of the initial material (n_0 = 3.36 for BZO_{50}) and the irradiated material ($n_k = 0.13\sqrt{k}$ for $NO_{50.k}$),
- $w_{el} \cdot 10^7$ – the intensity of the stress increase with unitary elastic deformation (=1.5 and 2.5 MPa / % of the initial material BZO_{50} and the irradiated material $NO_{50.k}$).

Lower values of the total deformation Z_{tot} and true plastic strain e_f in the compression test are visible in the irradiated material, in particular, when the stress exceeded 50 MPa (**Figs. 2** and **3**). The undesirable effect of irradiation is a slight decrease in elastic deformation, which drops by approximately one percentage point at stresses of $\sigma_{tot} \geq 100$ MPa (**Fig. 4**).

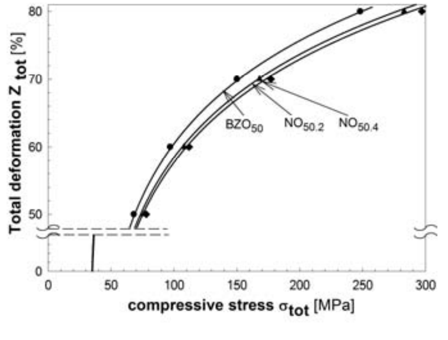


Fig. 2. Changes in total deformation during as a function of stress σ_{tot} (3)

Rys. 2. Zmiany całkowitej deformacji w funkcji naprężenia σ_{tot} (3)

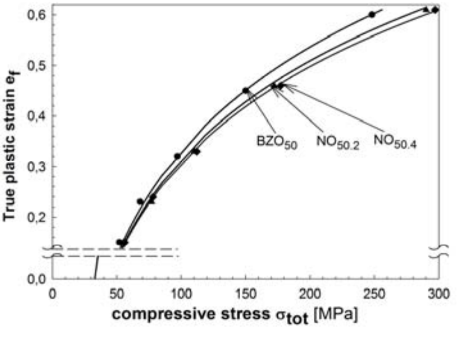


Fig. 3. Changes in true plastic strain during as a function of stress σ_{tot} (4)

Rys. 3. Zmiany rzeczywistego odkształcenia plastycznego w funkcji naprężenia σ_{tot} (4)

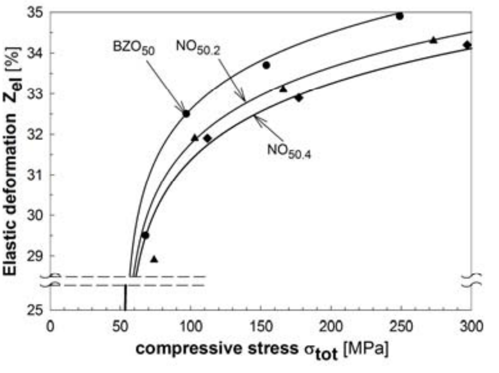


Fig. 4. Changes in elastic deformation during as a function of stress σ_{tot} (5)
Rys. 4. Zmiany odkształcenia sprężystego w funkcji naprężenia σ_{tot} (5)

The improvement of the resistance to plastic deformation was confirmed by hardness and Young's modulus tests. The *H* value increases with the *k* multiplication factor of the electron beam irradiation. It is observed that an increase in the degree of plastic deformation e_f during compression (which simulates operational deformation) is accompanied by a linear drop in hardness (**Fig. 5**) The changes in *H* were approximated (taking into account the measurement taken for BZ_k samples) by the regression equation (R.≥.0.95), (6):

$$H = H_{BZk} - I_H \cdot e_f \quad [MPa] \tag{6}$$

where: H_{BZk} – the hardness of the initial (base) material after being irradiated with an electron beam k-times **(Table 1),**

I_H – the intensity of the effect the true plastic strain e_f has on hardness (the change in hardness with a unitary increase of plastic deformation).

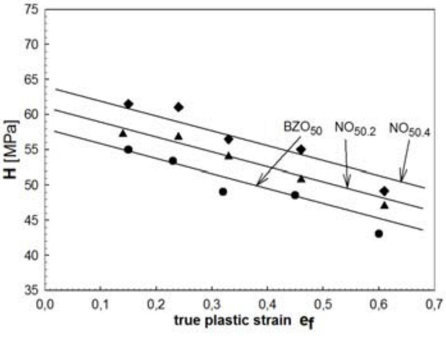


Table 1. The values of the equation (6)
Tabela 1. Wartości równania (6)

k	0	2	4
H_{BZk} [MPa]	58	61	64
I_H [MPa]	21		

Fig. 5. Changes in the hardness of the GUR 1050 polymer as a function of true plastic strain e_f (6)
Rys. 5. Zmiany twardości w polimerze GUR 1050 w funkcji rzeczywistego odkształcenia plastycznego e_f (6)

A similar co-dependence may be observed for the elasticity modulus *E*. It is a consequence of the linear co-relation of Young's modulus with hardness (7) with a co-relation ratio R = 0.96:

$$E = 25H - 350 \qquad [\text{MPa}] \qquad (7)$$

The analysis of the micromechanism and the wear resistance ratio suggests the increase in the tribological properties of the polymer after a radiation modification. Parameter β indicates that lower tendency of the carved material to chip rather than to uplift is maintained in all operational deformations e_f This proves the prevalence of ridging ($\beta \rightarrow 0$) over the micromechanism of wear through slicing ($\beta \rightarrow 1$), (**Fig. 6**). As a result, a low intensity of decrease in resistance to abrasive wear W_β is noted despite the increase in operational deformation e_f of the polyethylene. The increase in resistance W_β is proportional to the dose of the irradiation with an electron beam that GUR 1050 is subjected to (**Fig. 7**).

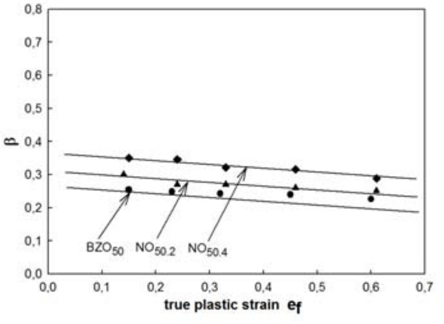


Fig. 6. Changes in abrasive wear micromechanism as a function of true plastic strain e_f

Rys. 6. Zmiany mikromechanizmu zużycia ściernego w funkcji rzeczywistego odkształcenia plastycznego e_f

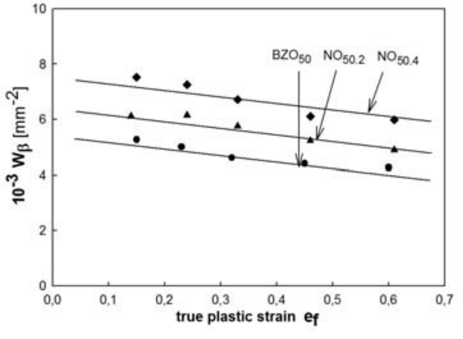


Fig.7. Changes in resistance to abrasive wear ratio as a function of true plastic strain e_f

Rys. 7. Zmiany wskaźnika odporności na zużycie ścierne w funkcji rzeczywistego odkształcenia plastycznego e_f

SUMMARY

The analysis of the changes in the deformation values being the result of operational load showed the increase in the resistance of polyethylene to total deformation as a result of applied irradiation with an electron beam. The improvement of its properties has been corroborated by the results of sclerometric and microindentation microtests. The increase in hardness and Young's modulus proportional to the dose of irradiation applied have been found. Scratch test showed the lower tendency of the modified polymer to chip rather than to uplift. This proves that the abrasive wear may result in the smaller loss of the mass of the material. After irradiation with an electron beam, a significant increase in wear resistance ratio is noted. Increasing operational deformation reduces the wear resistance of polyethylene.

The parameters of microtests may be used for the purpose of evaluation and selection of the polymers used in friction joints in medicine and technology.

The presented results will be subjected to verification in tribological tests of polymer and metal kinematic systems, which will reflect the conditions in which an endoprosthesis of a hip joint operates.

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Streszczenie

W celu podwyższenia odporności polietylenu GUR 1050 na odkształcenie plastyczne i zużycie ścierne zastosowano modyfikację radiacyjną. Badania w zakresie naprężeń do 300 MPa wykazały wzrost odporności na deformację trwałą. Potwierdzono to testami mikroindentacyjnymi i sklerometrycznymi. Scratch test wykazał, że w całym zakresie odkształceń eksploatacyjnych występuje mniejsza skłonność do wykruszania materiału wyżłobionego niż do jego plastycznego wypiętrzania. Dominacja mikromechanizmu bruzdowania pozwala zachować niewielki spadek odporności na zużycie. Parametry mikrotestów mogą być wykorzystywane do diagnozowania i selekcji polimerów stosowanych w medycznych oraz technicznych węzłach tarcia.