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THE EFFECT OF IRRADIATION WITH AN ELECTRON BEAM ON MECHANICAL, SCLEROMETRIC, AND TRIBOLOGICAL PROPERTIES OF PTFE WITH A GRAPHITE ADDITIVE

WPŁYW NAPROMIENIOWANIA STRUMIENIEM ELEKTRONÓW NA WŁAŚCIWOŚCI MECHANICZNE, SKLEROMETRYCZNE I TRIBOLOGICZNE PTFE Z DODATKIEM GRAFITU

Key words:

PTFE, modification with an electron beam, mechanical properties, wear

Słowa kluczowe:

PTFE, modyfikacja strumieniem elektronów, właściwości mechaniczne, zużycie

Abstract

Polytetrafluoroethylene (PTFE) with a 15% graphite additive was subjected to irradiation using an electron beam of energy of 10 MeV in doses of 26, 52, 78,

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104, and 156 kGy. The effect of electron beam irradiation on the mechanical, sclerometric, and tribological properties of the material was examined. It was found that the modification through irradiation entailed a gradual increase in the microhardness and Young's modulus as the absorbed dose of irradiation increased. A stereometric analysis of the scratch traces on the material allowed for the determination of the coefficients of the wear micromechanism, β, and resistance to wear, W_β. After irradiation (especially with a dose of 104 kGy), a significant quantity of the material showed traces of ploughing; the value of the wear resistance coefficient W_β for the PTFE subjected to cross-linking also increased considerably, which indicated a significant reduction of the tribological wear in relation to the initial material. Examination of abrasive wear of PTFE was performed for the polymer in its initial state and after modification through radiation on a pin-on-disc test stand, T-01. The tests have shown more than a five times reduction of the linear wear during the interaction with a titanium counter-specimen.

INTRODUCTION AND RESEARCH METHODOLOGY

Polytetrafluoroethylene (PTFE) belongs to the group of fluoropolymers, which have a number of desirable properties, such as the following: it has a relatively high thermal resistance – the temperature of long-term usage amounts to 260°C, high chemical inertness – it is insoluble in all known solvents, with an extremely low friction coefficient and resistance to atmospheric conditions. These properties contributed to the fact that this polymer is used in such market segments as electronics, electrical engineering, household appliances, medicine, as well as automotive, construction, and aerospace industries **[L. 1–5]**. Another characteristic feature of PTFE is its high density: 2.2 g/cm^3. A 15% graphite additive reduces the friction coefficient, improves rigidity and mechanical strength, and, to a lower degree, improves the resistance to tribological wear. PTFE without any additives is characterized by a very high wear intensity. It is possible to decrease the wear rate of the elements of bearings made of PTFE by three orders of magnitude, by adding different fillers. A reduction of the wear of non-modified PTFE in a pair with an interacting element with a smooth sliding surface may be further influenced by irradiation with an electron beam or with a gamma-ray beam **[L. 6–8]**.

Due to the limited availability of electron radiation sources with energy in the order of MeV necessary to penetrate PTFE to a depth of several centimetres, a majority of the available research results refer to the influence of gamma radiation **[L. 9]**. The research was usually conducted for PTFE without filler additives. The purpose of this study was to determine the effect of irradiation with an electron beam on the mechanical, sclerometric, and tribological properties of PTFE with a 15% graphite additive.

The research material was commercially available polytetrafluoroethylene (PTFE) with a 15% graphite additive (SM-G15, Inbras, Poland) in the form of rods, 20 mm in diameter. 20 mm high cylindrical specimens were cut out of the rods. The material in its initial condition was marked as BZ_{15}, while the irradiated specimens were designated as ($N_{15.i}$). The index 15 indicated the graphite content, the letter N showed the electron-beam irradiation, and *i* = 1-6 referred to the multiplication factor of the irradiation with a dose of 26kGy. Modification through irradiation was performed using a linear accelerator Elektronika 10/10 (energy of electrons: 10 MeV; beam power: 10 kW). In order to eliminate free radicals, the irradiated samples were stabilized through oxidation by means of thermal processing in a vacuum: heating to a temperature of 200°C for 4 hours, soaking for 2 hours, and cooling down to ambient temperature for 10 hours. Next, all the samples were vacuum wrapped.

The mechanical properties of the polymer in its initial state and after electron-beam irradiation were determined by means of the Micron-Gamma tester (manufactured by the Aviation Faculty, Technical University of Kiev), and a Vickers microhardness tester of Wolpert Wilson Instruments, model 401MVD. The load on the indenter during the tests of PTFE was 2.94 N, and the time of its work under full load was 10 seconds. The elasticity modulus, E, was determined with the standard Oliver-Pharr method **[L. 10]**; during measurements, a Berkovich penetrator was used, with 1 N load of the indenter and time of maximum loading of 15 s. After the approximation of the load removal curve with the second-degree polynomial, 70% of its scope was covered by the analysis. The measurement results were averaged for 7 indentations.

Sclerometric tests were performed with the Revetest Xpress device of CSM, using a Rockwell indenter (Y-275, 200 μm radius). During the scratch test, a normal force of 4 N and scratching rate of 5.4 mm/min were applied for a ca. 4 mm long scratch. The measurement of the furrow area *A* and the plastic elevation area *B* was taken by means of a Taylor Hobson profilographometer with the TalyMap Universal software. A scratch area of 2x2 mm was investigated, maintaining a sampling distance of x = 1 μm and y = 2 μm. In order to determine the coefficient of the polymers' resistance to tribological wear, W_β, 500 profilograms were subjected to an analysis. The value of W_β was calculated from formula **[L. 11]**:

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

where β_i – abrasive wear micromechanism determined from dependence (2) **[L. 12, 13]**:

$$\beta = \frac{1}{n}\sum_{i=1}^{n}\frac{A_i - B_i}{A_i} \qquad [-] \tag{2}$$

where A_i – furrow area, B_i – elevation area.

The tribological tests were made for the initial polymer and for the modified, i.e. irradiated polymer. In each case, 3 samples were prepared (in the forms of pins, 5 mm in diameter). Disc made of titanium Grade 2 were used as counter-specimens. The surfaces of the interacting elements were prepared so as to obtain a roughness in the order of Ra = 0.2 µm. This enabled the formation of a thin PTFE film which reduced wear. For the tribological tests, a pin-on-disc test stand T-01 (manufactured by ITeE Radom, Poland) was used. The tests were carried out under dry friction conditions. The pressure in the friction pair was 1 MPa (F_n = 20 N). The sliding rate in a one-way rotary motion was 0.1 m/s at a friction distance of 1000 m. Ambient conditions, a temperature of 21 ±1°C, and humidity of 50 ±5%, were in compliance with the recommendations of VAMAS **[L. 14]**. The linear wear L_w was determined as a difference between the indications of the micrometric sensor before and after the test (and after the cooling stage). The friction coefficient was determined as a quotient of the recorded friction force, F_t , and the normal force applied, F_n (20 N).

RESEARCH RESULTS AND THEIR ANALYSIS

The supramolecular structure of polymers, including the entanglement of macromolecules, has a strong impact on their properties. Segments of the chain trapped between the entanglements create a molecular network in which they function as physical crosslinks. In the case of PTFE, a destruction of polymer chains and the accompanying phenomenon of a large increase in the degree of crystallinity are the dominant effects of irradiation. Such a structure determines the course of a number of processes, as well as the properties of the polymer, e.g., the crystallization process, resistance to deformation, mechanical properties, or wear resistance.

The consequences of changes in the structure of PTFE with a 15% graphite additive induced by electron-beam irradiation include changes in the microhardness, *µHV*, and in Young's modulus, *E*. The course of these changes is presented in **Fig. 1**. The results the data indicate that the protective role of modification through irradiation against the effects of operational load finds its corroboration in the changes of the polymer's hardness and of the Young's modulus. Both these parameters increased considerably with the increasing irradiation dose absorbed, especially in the range of 52-156 kGy.

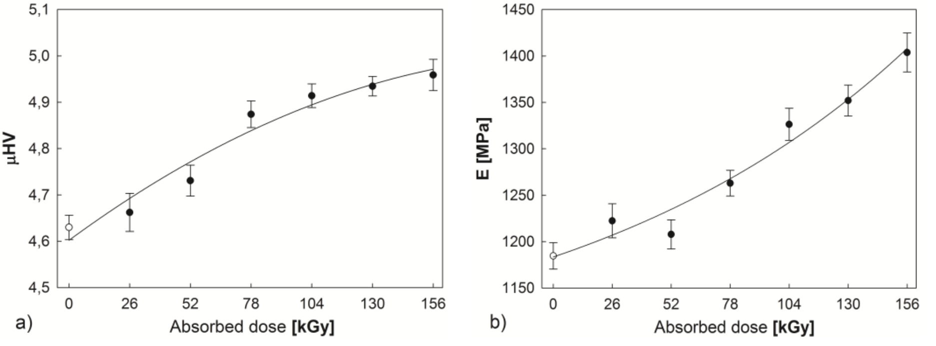


Fig. 1. **Changes in microhardness µHV (a), and Young's modulus E (b) as a function of electron-beam irradiation**

Rys. 1. Zmiany mikrotwardości µHV – a) i modułu Younga E – b) w funkcji napromieniowania strumieniem elektronów

The influence of irradiation on the PTFE scratch test parameters is shown in **Fig. 2**. Parameter *β*, determined based on a stereometric analysis of scratch traces (**Fig. 2a**), shows that a component of the ploughing micromechanism ($\beta \rightarrow 0$) predominated in the polymer subject to electron-beam irradiation. This means that a larger part of the irradiated material (in particular, with the dose of 104 kGy) underwent plastic deformation during the scratch test and was elevated on the edge of the scratch formed. The abrasive wear resistance ratio, W_β (**Fig. 2b**), increased for the doses of 26-104 kGy, while the 156 kGy dose caused an intensive reduction of the value of this ratio, which meant that such a dose led to degradation of the material.

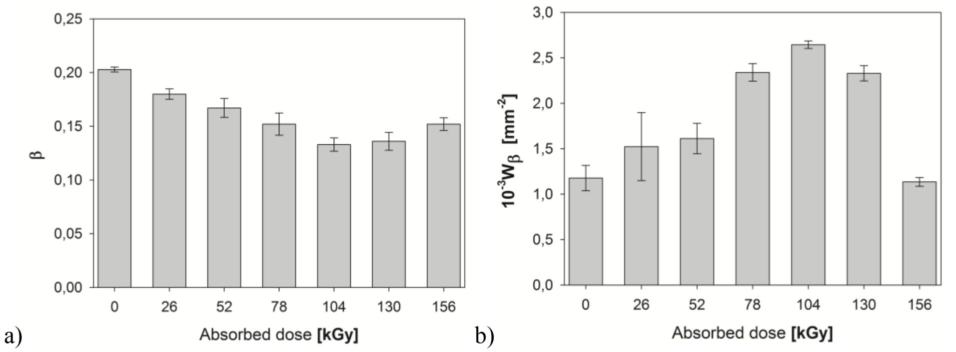
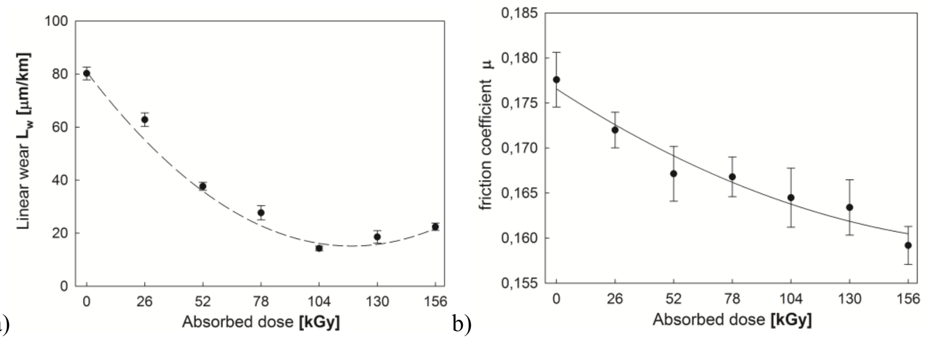


Fig. 2. The effect of electron-beam irradiation on the micromechanism of PTFE wear, β (a), and the wear resistance ratio, W_β – (b)

Rys. 2. Wpływ napromieniowania strumieniem elektronów na mikromechanizm zużycia PTFE β – a), oraz wskaźnik odporności na zużycie W_β – b)

A significant reduction in the linear wear rate, L_w, is observed as the irradiation dose absorbed by PTFE increases during the interaction with the titanium counter-specimen (**Fig. 3a**). The linear wear, Z_l (tester T-01) for the unmodified polymer BZ_{15} amounted to 80.25 μm/km, and for the polymer irradiated with a dose of 104 kGy, 14.25 μm/km, and this testifies to a more than a five times reduction in the wear of the polymer as a result of irradiation. Irradiation with a larger dose caused degradation of the material and another increase in wear. There was also a significant decrease of the friction coefficient in the material subjected to irradiation (**Fig. 3b**).



a) b)

Fig. 3. Linear wear (a), and friction coefficient μ (b) for PTFE during the interaction with titanium Ti Gr2

Rys. 3. Zużycie liniowe – a) i współczynnik tarcia μ – b) dla PTFE przy współpracy z tytanem Ti Gr2

CONCLUSIONS

- Improvement of the mechanical properties of polytetrafluoroethylene as a result of radiation modification was substantiated with the results of microtests. It was found that the microhardness and Young's modulus increased proportionally to the applied radiation dose.
- Stereometric analysis of the scratched surface of PTFE subjected to electron-beam irradiation showed a change of the wear mechanism β in the direction of ploughing, and it showed an increase in the resistance to abrasive wear, W_β, in particular for the absorbed dose of 104 kGy.
- Tribological tests showed more than a fivefold reduction of the intensity of linear wear and a reduction of the friction coefficient. It was also found that irradiation of PTFE with a dose of more than 104 kGy caused its degradation and an increase of its linear wear.

- The obtained research results prognosticate a longer life of the modified polymer in the sliding components of bearings, which do not require lubricating, e.g., in air compressors.

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Streszczenie

Politetrafluoroetylen z 15% dodatkiem grafitu poddano napromieniowaniu strumieniem elektronów o energii wiązki 10 MeV, w dawkach 26, 52, 78, 104, 156 kGy. Zbadano wpływ napromieniowania na właściwości mechaniczne, sklerometryczne i tribologiczne tworzywa. Stwierdzono, że modyfikacja radiacyjna powoduje stopniowe podwyższenie mikrotwardości oraz modułu Younga w miarę wzrostu pochłoniętej dawki napromieniowania. Stereometryczna analiza śladów zarysowania materiału umożliwiła wyznaczenie wskaźnika mikromechanizmu zużycia β i odporności na zużycie $W_β$. Po napromieniowaniu (zwłaszcza dawką 104 kGy) znacząca ilość materiału ulega bruzdowaniu; silnie rosła także wartość wskaźnika odporności na zużycie $W_β$ PTFE poddanego sieciowaniu, co wskazywało na znaczne ograniczenie zużycia tribologicznego w stosunku do materiału wyjściowego. Badania zużycia ściernego politetrafluoroetylenu wykonano dla polimeru w stanie wyjściowym oraz po zmodyfikowaniu radiacyjnym na stanowisku T-01 w skojarzeniu trzpień–tarcza. Testy wykazały ponadpięciokrotne zmniejszenie zużycia liniowego podczas współpracy z tytanową przeciwpróbką.