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# THE EFFECT OF THE IONIZING RADIATION ON THE TRIBOLOGICAL PROPERTIES OF THERMOPLASTIC POLYMERS RUBBING AGAINST STEEL

# WPŁYW DAWKI PROMIENIOWANIA GAMMA NA WŁAŚCIWOŚCI TRIBOLOGICZNE WYBRANYCH POLIMERÓW TERMOPLASTYCZNYCH WSPÓŁPRACUJĄCYCH ZE STALĄ

## Key words:

gamma irradiation, PEEK, PEI, PET, PA6, microhardness, friction coefficient

## Słowa kluczowe:

promieniowanie gamma, PEEK, PEI, PET, PA6, mikrotwardość, współczynnik tarcia

#### Abstract

The paper describes the effects of gamma radiation doses on the tribological and mechanical properties of selected thermoplastic polymers: PEEK, PEI, PET, and PA6. Samples of those polymers were irradiated with gamma rays of energy 1, 1.2, 3, 4, 5 MeV and with the intensity of the radiation beam 4, 7, 10,

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30, 40  $\mu$ A. With an increasing dose of radiation, properties such as microhardness, the friction coefficient of polymers, and the wear rate are changed. The chemical structure of the irradiated polymer may be changed by crosslinking but also by chain scission. The modified polymers may be used in medical science, electronics, and space technology.

In this article, we report the results of steel C45 – thermoplastics (PEEK, PEI, PET, and PA6) friction couple during the slip motion. The analysis is focused on the friction coefficient, wear rate, and microhardness. Kinematic parameters of the experiment in slide movement were constant and defined by the following values: contact pressure p = 1MPa and sliding velocity v = 1m/s. The authors observed that, as gamma radiation dose increased, the microhardness and friction coefficient increased and their wear intensity decreased. Polymers change their microstructure and molecular structure when they are subjected to gamma ray irradiation, which results in modification on many important physical and chemical properties.

## **INTRODUCTION**

Thermoplastics, e.g., PEEK, PEI, PET, and PA6, are one of the most promising polymers. They are widely used for various applications because of their mechanical properties, e.g., in machine building as elements of high dimensional stability, that are load and wear resistant (e.g. bearings) [L. 1]. Apart from their good tribological properties, some engineered polymers, e.g., PEEK and PEI, have good ductility, formability, and light weight. It is making them an obvious choice for replacing ceramic and metallic components [L. 2]. Due to theirs excellent mechanical and physical properties, they are used in a wide range of applications such as electronics, transportation, energy industrial, and semiconductors [L. 3].

The radiation dose required to bring major changes in the morphological structure of the polymer, and in the appearance, chemical and physical states, and mechanical, tribological, electrical, and thermal properties **[L. 4, 5, 6]**.

Research into the effects of high-energy, ionizing radiation on polymers is receiving increased attention from several points of view:

- The development of radiation as a tool for basic polymer research;
- The useful modification of polymers by controlled irradiation; and,
- The development of radiation-resistant materials.

Of vital importance to all of these areas is an understanding of the basic radiation chemistry that occurs in each specific polymer.

Ionizing radiation affects the chemical structure and properties of polymers. The primary factor that determines the direction of radiation effect is the chemical structure of the polymer.

The changes in physical properties of a polymer under ionizing radiation will depend primarily on whether the polymer chains are crosslinked or whether they are degraded. When a polymer is exposed to ionizing radiation, it undergoes scission, crosslinking, and hydrogen evolution, and these events have deleterious effects on many physical and chemical properties. Crosslinking produces an increase in modulus and a decrease in elongation, while degradation causes decreases in modulus and strength. Oxidative degradation also has been observed in gamma polymers. Nonetheless, some polymers are thought to undergo some biodegradation due to oxygen radicals. According to basic polymer chemistry, oxidation can occur in a polymer if it is exposed to air during or after exposure to irradiation. Additionally, structural changes occurring within the polymer matrix due to irradiation may also adversely affect the mechanical properties.

Gamma irradiation treatment provides a unique way to modify the chemical, structural, optical, mechanical, and electrical properties of polymer by causing irreversible changes in their macromolecular structure [L. 7].

# MATERIALS AND MEASURING METHOD

Four thermoplastics were tested: PEEK, PEI, PET, and PA 6. These polymers are the most commonly used thermoplastic materials. Those polymers are semicrystalline polymers containing amorphous and crystalline phases (polymer chains in crystalline region are denser than are those on the amorphous region). Modification through radiation was performed in an ion beam radiation facility at D. Serikbaev East Kazakhstan State Technical University.

The samples were irradiated with a source of rays from a gamma isotope Cobalt-60 in the radiation chamber at room temperature and at ambient pressure, with the following doses of energy and the intensity of the radiation beam:

- ➤ 1 MeV, 30µA,
- ➤ 1.2 MeV, 30, 40 µA,
- ➤ 1.4 MeV, 30 µA,
- ≥ 3 MeV, 7 μA,
- ≻ 4 MeV, 4, 7, 10 µA,
- ➤ 5 MeV, 7 µA.

The polymers samples of size 8 mm length and 30 mm diameter were cut from the commercially available materials and were used without any further treatment. One sample was kept as a control and the other samples were subjected to irradiation.

The effect of different energy ion beam irradiations on the mechanical and tribological properties were studied. The gamma irradiation has been found to have the ability to expose the whole area of the sample; therefore, it was expected to create homogenous modification in it.

Tribological properties such as friction coefficient, linear tribological wear, and friction force were measured by using *pin-on-disc* tribometer T-01 M

(MCNEMT, Radom) **[L. 8]**. Measurements were carried out at room temperature in ambient conditions. The change in mechanical and tribological properties induced by irradiation of 1 MeV and 5 MeV were studied for PEEK, PEI, PET, and PA6, also for non-irradiated sample of those polymers.

Samples cooperated during the test with a flat surface made of steel C45, the surface of which was mechanically polished and had an average of the profilometric parameter of roughness  $Ra = 0.6 \mu m$  and the hardness of 40 HRC. The geometrical dimensions of the steel roll were diameter = 38 mm and width = 5 mm. PEEK, PEI, PET and PA6 pin samples were held by a pin holder against the rotating disc mounted on the disc holder. Before each test, the polymer pin and C45 stainless steel discs were thoroughly cleaned with alcohol/acetone. The tests were conveyed on a 5-kilometre path of friction with kinematic parameters defined by the following values: contact pressure p = 1 MPa, sliding velocity v = 1 m/s, with an initial phase of 30 min grinding with surface thrust of 0.5 MPa, and an average sliding velocity v = 1 m/s. The normal force for all the cases was constant at  $F_n = 1$  MPa. The sliding distance of 5 km was kept constant for all tests. The geometry of the contacting surfaces was constant. During the experiment, friction force  $F_t$  was recorded with an analogue-to-digital converter. A plot of the variation of friction coefficient with varying sliding distance was displayed online during the test. After stabilization of the plot, the average value of friction coefficient was reported. Wear of the pin was evaluated by measuring length loss after completing the test.

Moreover, microhardness measurements were performed using Vickers's method. Measurement HV 0.1 microhardness was carried out using a Micro Hardness SHIMADZU hardness tester. The load was 1 N with a dwell time of 30 s. These tests were carried out at room temperature. To find accurate results, at least six readings at different locations on the samples under the same conditions were determined and average values were reported.

#### **RESULTS AND DISCUSSION**

Measurements of microhardness values of polymers irradiated with gamma rays and standard deviations ( $\sigma$ ) were calculated for each series of measurements. On the surface of each polymer sample, areas that collaborated with the steel plate were defined, and six measuring sections were selected. The results of measurements on the polymers are summarized in **Table 1**.

**Table 1** presents the results of Vickers microhardness measurements conducted on the studied materials irradiated with gamma rays. Obtained results indicate that an increase of radiation doses from 1 MeV to 5 MeV does not cause any significant changes in the microhardness of the studied polymers.

The highest microhardness (27.42 HV 0.1) value was obtained for PEEK with radiation dose of 1.2 MeV (40  $\mu$ A). PEEK microhardness value increases between two radiation doses – with doses ranging from 1 MeV to 1.2 MeV

amounting to about 10% and from 1.4 MeV to 4 MeV amounting to about 15%. For this polymer, microhardness values decrease between radiation dose from 4 MeV (10  $\mu$ A) to 5 MeV and a very low microhardness value was measured with a radiation dose of 5 MeV (17.62 HV 0.1). For PA6, the lowest microhardness (9.4 HV 0.1) value was measured at 4 MeV (4  $\mu$ A), and the greatest microhardness (13.84 HV 0.1) value was obtained at 1.2 MeV (30  $\mu$ A). Further increasing the radiation dose caused an increase in microhardness results with doses ranging from 1 MeV to 1.2 MeV (30  $\mu$ A). The differences between the results are more than 35%. The materials irradiated with energy equal to 4 MeV (4  $\mu$ A) are characterized by lower microhardness values due to the measurement of microhardness with an increase in the intensity of radiation 10  $\mu$ A from 3 to 7%. For only PA6, the result measurement of microhardness for those doses increase approximately to 14%.

Table 1.	The results of measurements of microhardness parameter of polymers irradiated
	with gamma rays
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Tabela 1. Zestawienie wyników pomiaru mikrotwardości oraz odchylenia standardowego (σ) dla polimerów napromieniowanych

Dose		PEEK	PEI	PET	PA6
1 MeV	microhardness [HV 0.1]	25.03	21.67	17.47	11.79
30 µA	σ[HV 0.1]	4.08	1.15	0.66	2.11
1.2 MeV	microhardness [HV 0.1]	26.98	22.33	20.42	13.84
30 µA	σ[HV 0.1]	1.24	1.67	1.3	2.05
1.2 MeV	microhardness [HV 0.1]	27.42	26.0	18.29	10.12
40 μΑ	σ[HV 0.1]	5.48	2.23	3.15	0.89
1.4 MeV	microhardness [HV 0.1]	22.85	23.03	18.63	12.50
30 µA	σ[HV 0.1]	1.12	4.41	1.69	2.37
3 MeV	microhardness [HV 0.1]	24.42	26.16	18.2	12.14
7 μΑ	σ [HV 0.1]	1.69	4.13	1.38	1.27
4 MeV	microhardness [HV 0.1]	24.02	23.40	17.07	9.4
4 μΑ	σ[HV 0.1]	2.51	4.27	1.31	0.85
4 MeV	microhardness [HV 0.1]	26.31	21.15	19.4	11.02
7 μΑ	σ[HV 0.1]	1.37	1.22	1.39	1.58
4 MeV	microhardness [HV 0.1]	22.36	21.68	16.52	10.92
10 µA	σ[HV 0.1]	3.13	2.76	1.77	2.58
5 MeV	microhardness [HV 0.1]	17.62	22.62	16.88	10.46
7 μΑ	σ[HV 0.1]	3.5	1.74	1.58	1.88

It can be concluded from analysis of obtained results that an increase in radiation dose increases the microhardness of the studied materials, depending on the radiation dose. Because the measurements performed on the data are in

the narrow range of radiation doses, there are no explicit rules about how the radiation dose influences the mechanical properties of the studies materials. Further research on materials for a wider range of radiation doses would allow us to better define the relationship between the mechanical properties of polymers exposed to gamma radiation.

The microhardness measurements were taken for non-irradiated sample in order to compare the effect of radiation dose from 1 MeV to 5 MeV on mechanical properties of polymers. The measurement results are summarized in **Tab. 2** and illustrated by the graph in **Fig. 1**.

Table 2.	The results of	measurements	of the	microhardness	parameter	of polymers	not
	irradiated						

	PEEK	PEI	PET	PA6
microhardness [HV 0.1]	21.82	13.17	14.92	11.95
standard deviation $[\sigma]$	2.46	0.63	0.79	1.3

Tabela 2. Zestawienie wyników pomiaru dla polimerów nienaświetlonych

Obtained results in **Table 1** and **2** indicate that an increase in gamma radiation dose causes an increase in microhardness values by 1.98-12.99 HV 0.1 units (depending on the studied materials). The greatest increase in microhardness by 12.99 HV 0.1 units was achieved for PEI (from 13.17 to 26.16 HV 0.1), and the lowest increase by 1.89 HV 0.1 units was achieved for PA6 (from 11.95 to 13.84 HV 0.1).

Based on the conducted studies, it was stated that materials treated with gamma radiation are characterized by higher values of microhardness than the polymers that do not undergo this type of modification.

Polymers irradiated with gamma radiation can be crosslinked, which results in an increase in the hardness of the modified materials. In further studies, structural aspects of aromatic polymers will be analysed using X-ray diffraction techniques. These techniques can indicate that the crystallinity increases with the increase of dose. When the semi-crystalline polymers are exposed to radiation, the crystalline lamellas may be broken into two or more portions, whereas the long molecular chains of amorphous regions may be broken at different places leaving a free smaller chain, which immediately comes to a stable position by collecting its whole length into a regularly arranged lamella form. Further materials research could confirm this assumption.





Rys. 1. Zmiana mikrotwardości [HV 0,1] dla polimerów napromieniowanych oraz niepoddanych modyfikacjom

Figure 1 shows the dose dependence of microhardness change for nonirradiated samples and samples irradiated with doses ranging from 1 MeV to 5 MeV. It is seen from Fig. 1 that, for a given dose, the microhardness increases with the increase in the dose of radiation. The control samples (not irradiated) exhibit the lowest microhardness values for two polymers: PEI and PA6, the greatest for PEEK. PEI and PET polymers are characterized by the greatest microhardness measurements values. The smallest influence of gamma radiation on the microhardness is characterized by PA6. According to **Tab. 1** and Tab. 2, the microhardness increases rapidly with dose in the range from 1 to 1.2 MeV. However, in our studies in irradiated polymers, we observed microhardness values that decrease with increasing gamma dose. Results showed that microhardness of irradiated PA6 at all the selected doses decrease approximately to 21%, as compared to the unmodified material. No plateau was observed in the studied materials. Comparing the results of measurements for irradiated polymers and non-irradiated polymers, it can be observed an increase microhardness values. This implies that gamma irradiation enhance mechanical properties of polymer.

The authors attribute this to crosslinking that increases with radiation dose. Whether hardness values increase or decrease depends on the amount crosslinking and/or scission on changes in crystallinity. With this in mind, the microhardness data is shown in **Fig. 1** and explained by defect kinetics. These defects can arise from chain scission and crosslinking in crystallinity, etc.

depending on the types of defects, changes in one or more physical, mechanical, and chemical properties will be noted. Polymers change their microstructure and molecular structure when they are subjected to gamma ray irradiation. Both microstructure and molecular structure after gamma radiation are unstable and radiation induces changes that result in more stable matrices. Whether hardness values increase or decrease depends on the amount crosslinking and/or scission, on changes in crystallinity, and on the degree to which the matrix undergoes physical aging in amorphous regions during annealing.

The reason for increasing the microhardness materials may be the changes in the chemical structure of polymers that include crosslinking process, which causes curing of the polymer. With the increase in the dose of gamma radiation, further changes properties of materials are possible, e.g. an increase in their fragility. This type of accuracy has been described in [L. 9], and the studied material was polymer PE-UHMW. This study investigates the generation of radiation and annealing induced structures that influence the hardness of highdensity polyethylene (HDPE). Hardness increases with increasing gamma ray dose, annealing temperature, and annealing time. In the stated literature, it was observed that the increase in radiation dose of 5 to 10 times the hardness of the polyethylene increases to 23%, but this entails higher brittleness and susceptibility to fracture.

The change in mechanical and tribological properties induced by irradiation of 1 MeV and 5 MeV were studied for PEEK, PEI, PET, and PA6, also for non-irradiated sample of those polymers. The friction and wear tests were conducted on a *pin-on-disc* test rig. The measurement results are summarized in **Tab. 3** and illustrated by the graph in **Fig. 2**.

Table 3.	The results of measurements of coefficient friction of polymers irradiated with
	gamma rays of energy 1 MeV and 5 MeV at the defined values of motion parame-
	ters: contact pressure $p = 1$ MPa, sliding velocity $v = 1$ m/s

	PEEK	PEI	PET	PA6
$\mu$ polymers irradiated with 1 MeV	0.304	0.379	0.232	0.994
$\mu$ polymers irradiated with 5 MeV	0.397	0.329	0.272	1.096
$\mu$ polymer non-irradiated	0.289	0.392	0.343	0.264

Tabela 3. Wartość współczynnika tarcia (μ) dla polimerów napromieniowanych energią 1 MeV i 5 MeV, parametry tarcia: siła nacisku p = 1 MPa, prędkość ślizgania v = 1 m/s

The influence on the applied modification on the friction coefficient value of the slide pair and on the wear intensity of the materials analysed were determined based on the obtained data from the tribological test. According to **Tab. 3**, the lowest friction coefficient among irradiated materials was measured for PET for both doses of radiation: 1 and 5 MeV. The greatest parameter was

measured for PA6. The highest friction coefficient was depicted by samples irradiated at a 5MeV irradiation dose. For the friction couple C45–PA6 (irradiated with 5 MeV), the friction coefficient increased by approximately 1.096 compared to other cases. The source of the increase has not been definitely identified, but it may be caused by the local temperature increase (the temperature of layers increase and the temperature inside the polymer). It may cause that the matrix becomes softer and will change its physical state (glass transition temperature for PA6 is  $47^{\circ}$ C).

From the observed dependencies, it can be concluded that the radiation modification of 1 to 5 MeV for the studies' materials caused an increase in the friction coefficient. A slight decrease in this ratio occurred during the test for PEI and PET. In order to compare effects of irradiation on tribological properties for the studied materials, tribological tests were also performed on samples non-irradiated for the same conditions. Almost for all non-irradiated materials, friction coefficient was lower. Changes in the coefficient of static friction may result from structural changes in polymers, e.g., as a result of crosslinking. These materials in the future will be examined for changes in their structures to be able to accurately determine the effect of gamma radiation on the physicochemical properties of polymers. **Figure 2** presents the variation of coefficient friction with change in radiation doses.





Rys. 2. Zmiana współczynnika tarcia ( $\mu$ ) dla polimerów napromieniowanych 1 MeV i 5 MeV, parametry tarcia: siła nacisku p = 1 MPa, prędkość ślizgania v = 1 m/s

After the process of friction, the microhardnesses were measured for the studied materials. The results of microhardness and the intensity of tribological wear of the friction process are summarized in **Table 4**.

# Table 4.The results of measurements of hardness parameter of polymers irradiated with<br/>gamma rays at the defined values of motion parameters: p = 1 MPa, v = 1 m/sTable 4.The results of measurements of hardness parameters: p = 1 MPa, v = 1 m/s

Tabela 4.	Zestawienie wyników pomiaru	dla polimerów	napromieniowanych	1 po	processe	tarcia
	dla parametrów ruchowych: p =	= 1  MPa, v = 1  m	n/s			

Dose		PEEK	PEI	PET	PA6
	microhardness [HV 0.1]	25.95	23.28	20.42	14.21
1 MeV	σ[HV 0.1]	0.46	1.14	3.06	0.65
30 µA	wear [mm]	0.05	0.739	0.01	0.012
	wear intensity [mm/km]	0.010	0.148	0.002	0.002
	microhardness [HV 0.1]	25.49	26.06	17.04	14.67
5 MeV	σ[HV 0.1]	1.04	2.26	0.52	1.38
7 μΑ	wear [mm]	0.087	0.347	0.01	0.044
	wear intensity [mm/km]	0.0174	0.0694	0.002	0.009
	microhardness [HV 0.1]	24.36	23.58	16.2	4.48
• • • •	σ[HV 0.1]	4.33	1	0.77	0.73
non-irradiated	wear [mm]	0.07	1.08	0.1	0.02
	wear intensity [mm/km]	0.014	0.216	0.020	0.004





Rys. 3. Zmiana intensywności zużycia dla polimerów napromieniowanych energią 1 MeV i 5 MeV, parametry tarcia: siła nacisku p = 1 MPa, prędkość ślizgania v = 1 m/s

**Figure 3** and **Table 4** show the intensity of tribological wear of the studied polymers recorded before (non-irradiated) and after irradiation. The tribological research was done in the technically dry friction conditions on the testing machine of the *pin-on-disc* T-01 M. The research subject matter of the authors concerns the comparison of the strengthening effects of the thermoplastics irradiated with gamma rays.

The lowest value of the linear wear was observed in the PET after irradiation for both gamma radiation dose. For non-irradiated sample, the lowest value of the linear wear was observed for PA6 (5 times less than for PET). The largest linear wear before and after irradiation was shown by the PEI, and it was found to decrease significantly with radiation exposure. Such a significant decrease in the tribological wear intensity of the polymer can be the reason of the increase of the microhardness. The material loss of the surface layer of the steel in the tribological sample was caused by the adhesive wear.

Wear and friction are surface phenomena, and modification of surface leads to change in wear and friction mechanisms. The wear rate may change with either change in hardness or in surface conditions. In general, increased hardness is responsible for increased wear resistance of a material. In current work, the hardness of irradiated material at all the selected doses is approximately the same; however, the wear rate is different. The reason behind this may be that, as the degree of crosslinking is changing, surface conditions are also changing.

Figure 3 indicates that gamma radiation dose plays a significant role in the strengthening of the material studied. Radiation modification can be one of the methods of strengthening the surface layer of the thermoplastics. Irradiated polymers characterized by greater resistance to wear and were harder than were their unmodified counterparts. The higher the radiation dose, the lower was tribological wear intensity. The results indicate a significant effect of the dose of gamma rays on tribological and mechanical properties of the tested materials. Analysis of the results suggests that, under the influence of radiation modification, improvement may be made on the coefficients friction [L. 4, 10] intensity of tribological wear [L. 9, 11]. The research presented in the paper [L. 2], showed that, with increasing microhardness, the material subjected to gamma radiation has increasing wear resistance. The value of microhardness increase is because of friction, but this increase is temporary, which is discussed in [L. 12].

In a further process of exploitation, hardness decreases, which is due to material losses arising as a result of fatigue wear. Fatigue wear is caused by contact between asperities with very high local stress and are repeated during sliding with or without lubrication. Microhardness measurement can then be used to determine the degree of fatigue damage in parts of machine parts.

# SUMMARY AND CONCLUSIONS

The results of the research prove the positive impact on the micro-hardness, wear rate, and friction coefficient of gamma irradiation on the mechanical and tribological properties of selected thermoplastic polymers. The observed increase of microhardness and friction coefficient related to the decrease in wear rate can be related to the irradiation of the polymers. Based on conducted studies, it was stated that as radiation doses increases, so too does the microhardness and coefficient of friction of the studied polymers. As the radiation dose increases, so does the microhardness of polymers. For comparison, the effect of the radiation modification of the properties of the material in the article presents the results of measurements of an unmodified polymer.

Gamma ray can produce changes in the semi-crystalline structure that may affect the properties of the polymer, e.g., an increase in hardness, mechanical strength, or heat resistance. This may be due to degradation, scissioning, and crosslinking of the polymer chains with increasing dose.

The changes in physical properties of polymers under ionizing radiation may be the result of crosslinking or scission of the polymer molecules. Polymers containing aromatic groups are usually the most radiation resistant.

In further studies (by FTIR spectroscopy and XRD), the structure of materials under ionizing radiation will be determined in order to confirmed observed changes in the mechanical and tribological properties. The effect of gamma irradiation on the structural properties on polymer samples will be analysed using conventional techniques, which are used to detect radiation-induced defects in the studied materials. FTIR and XRD have been used extensively to study radiation effects on polymers. We confirmed that with increase in the absorbed dose would increase the crystallinity and crystallite size and also increase the formation of defects in the materials. It can be concluded that the gamma irradiation of polymers is a suitable method for modification in material properties.

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#### Streszczenie

Celem niniejszej pracy było określenie wpływu dawki promieniowania gamma na właściwości tribologiczne i mechaniczne wybranych polimerów termoplastycznych: PEEK, PEI, PET, PA6. Wraz ze wzrostem dawki tego promieniowania właściwości takie jak microhardness, współczynnik tarcia statycznego oraz intensywność zużycia ulegają zmianie. Wymienione właściwości mogą być modyfikowane na skutek zachodzących zmian w strukturze chemicznej naświetlonych polimerów poprzez sieciowanie łańcuchów polimerowych, ale także ich rozerwanie. Zmodyfikowane polimery mogą być wykorzystane m.in. w medycynie, elektronice i technologii kosmicznej. W pracy opisano badania par ślizgowych stal C45 - wybrany polimer. Analizie poddano wartości współczynnika tarcia statycznego oraz intensywności zużycia materiału. Dla wszystkich analizowanych przypadków wartości parametrów ruchowych były stałe: siła nacisku wynosiła 1 MPa, a prędkość ślizgowa 1 m/s. Eksperyment wykazał zwiększenie wartości mikrotwardości i współczynnika tarcia oraz zmniejszenie intensywności zużycia.