Abstract

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TRIBOLOGICAL CHARACTERISTICS OF SELECTED MATERIAL PAIRS INTERACTING IN AIR AND VACUUM

CHARAKTERYSTYKA TRIBOLOGICZNA WYBRANYCH PAR MATERIAŁÓW ODDZIAŁUJĄCYCH W POWIETRZU I W PRÓŻNI

Key words: friction, wear, PVD coatings, vacuum.

The study examined the tribological characteristics of a ball-on-disc type sliding friction joint operating in air and in a vacuum under conditions of technically dry friction. The test elements included discs made of 100Cr6 steel without coating, with an anti-wear TiN coating, and a low-friction MoS_2/Ti coating, and bearing balls made of 100Cr6 steel and Al_2O_3 as counter-samples. The tribological properties of the studied friction joint were evaluated based on changes in the coefficient of friction, the intensity of linear wear, the ball wear diameter, and the appearance of worn surfaces. The environment of the friction joint was found to have a significant effect on the tribological characteristics of the studied friction pairs.

Słowa kluczowe: tarcie, zużycie, powłoki PVD, próżnia.

Streszczenie Zbadano charakterystyki tribologiczne ślizgowego węzła tarcia typu kula–tarcza pracującego w powietrzu oraz próżni w warunkach tarcia technicznie suchego. Elementami testowymi były tarcze wykonane ze stali 100Cr6 bez powłoki i z naniesionymi powłokami: przeciwzużyciowąTiN i niskotarciową MoS₂/Ti oraz kulki łożyskowe ze stali 100Cr6 i Al₂O₃ jako przeciwpróbki. Właściwości tribologiczne badanego węzła tarcia oceniano na podstawie przebiegu zmian współczynnika tarcia, intensywności zużywania liniowego, średnicy wytarcia kulki oraz wyglądu wytartych powierzchni. Wykazano istotny wpływ otoczenia węzła tarcia na charakterystyki tribologiczne badanych skojarzeń ciernych.

INTRODUCTION

The requirements applicable to friction joints operating in a vacuum environment are usually considerably more rigorous than in the case of friction joints working in air under normal atmospheric conditions. In vacuum conditions, there is contact between physically clean surfaces, which may exhibit strong adhesive bonds potentially leading to high friction resistances and a high degree of wear, particularly in sliding contacts [L. 1].

The contact between physically clean surfaces can also accompany friction in other environments, e.g., in dry or mixed friction conditions, when the oxide layers break up in the contact micro-areas or the lubricant film ruptures, or during machining, when a physically clean surface is exposed as a result of the separation of the machined material.

Understanding the phenomena and processes occurring during the friction of elements with physically clean surfaces requires tests conducted under vacuum conditions. On their basis, it will be possible to select appropriate materials for the interacting elements and technologies for their surface layers.

The latest research trends aimed at achieving appropriate tribological properties in joints operating in challenging environmental conditions and at high loads focus on anti-wear and anti-friction coatings [L. 2-6]. Anti-friction coatings can act as a solid lubricant, enabling the joint to operate under conditions

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oxide Al₂O₂.

METHODOLOGY

samples (bearing balls) of 100Cr6 steel and aluminium

The tests were carried out in a test station T-26

(Fig. 1a) developed by ITeE – PIB in Radom (Poland),

and involved test runs of a friction pair consisting of

a fixed ball pressed against a disc making rotary motion

(Fig. 1b) operating in air (surrounded by air) and in

a vacuum, under conditions of technically dry friction.

of technically dry friction, which is highly significant in the vacuum environment [L. 1, 6, 7].

The aim of the studies presented in this article was to determine and compare the tribological characteristics of a ball-on-disc type sliding friction joint operating in air or in vacuum under conditions of technically dry friction. The tested friction pair consisted of samples (discs) made of 100Cr6 steel without coating, with an anti-wear coating of titanium nitride TiN and a low-friction coating of titanium-doped molybdenum disulphide MoS_2/Ti (trade name MoST), and counter-



Fig. 1. Testing device and test conditions: a) view of the device; b) schematic representation of the friction joint Rys. 1. Urządzenie badawcze i warunki badań: a) widok urządzenia; b) schemat węzła tarcia

In each test run, at the set values of load F and sliding velocity V_{s} , continuous measurements of friction force F_t and total linear wear of the friction elements were performed. Based on data obtained from the test runs, the values of coefficient of friction μ and intensity of wear were determined. Parameters controlled during the test run included pressure p in the vacuum chamber and rotational velocity of the disc n which was calculated on the basis of sliding velocity V_s and friction radius. The number of rotations made by the disc was selected in such a way that friction distance, regardless of friction radius, was the same and equal to 1,000 m. The diameter of the balls interacting with the discs was 10 mm. Discs made of 100Cr6 steel were coated with a standard anti-wear coating of titanium nitride (TiN), with a thickness of 1.7 μ m, a hardness of 1,800 HV, and a surface roughness characterized by the parameter Ra = 0.35 μ m, and a low-friction coating of titanium-doped molybdenum disulphide (MoS₂/Ti) with a thickness of 1.7 μ m, hardness of 400 HV and surface roughness Ra = 0.33 μ m. The basis of reference was a pair in which the disc was made of 100Cr6 steel heat treated to a hardness of 62±2 HRC. After the heat treatment, the steel discs were ground and polished to achieve surface

roughness $Ra = 0.17 \mu m$. Both coatings were applied in the process of PVD (Physical Vapour Deposition). Before each test run, the samples (ball and disc) were washed for about 10 min in an ultrasonic cleaner in isopropyl alcohol and then dried in a stream of warm air. At least 3 test runs were carried out for each material pair.

The following pairs were studied in the ambient atmosphere (air) and in vacuum: 100Cr6–100Cr6, 100Cr6–Al₂O₃, TiN–100Cr6, TiN–Al₂O₃, MoS₂/Ti– -100Cr6, and MoS₂/Ti–Al₂O₃. The tribological properties of the studied pairs were evaluated based on changes in the coefficient of friction (COF) and intensity of linear wear, as well as the diameter of ball wear and the appearance of the geometric surface structure (GSS) of wear tracks determined using an interferometric profilographometer and an optical microscope.

RESULTS AND DISCUSSION

Examples of changes in COF and total linear wear for the elements of the model friction joint as a function of distance, determined for the material pairs 100Cr6–-100Cr6, TiN–100Cr6, and MoS_2/Ti –100Cr6 operating under conditions of technically dry friction in air and in vacuum, are shown in **Figs. 2–4**, respectively.

When analysing the graphs in **Fig. 2** generated for the pairs in which the disc was made of 100Cr6 steel, it can be seen that both the changes in COF and wear intensity depend on the environment and the material of the interacting elements.

The graphs demonstrate that, in the case of friction in air, in an environment consisting of oxygen, nitrogen and moisture (water vapour), at a constant sliding



Fig. 2. Changes in the coefficient of friction and total linear wear in the pair of counter-samples of 100Cr6 steel and Al₂O₃ with a disc of 100Cr6 steel

Rys. 2. Przebieg zmian współczynnika tarcia i sumarycznego zużycia liniowego skojarzenia przeciwpróbek ze stali 100Cr6 i Al₂O₃ z tarczą ze stali 100Cr6

velocity and constant normal load, the COF of the pair 100Cr6–100Cr6 is stable over the whole friction distance and its mean value is approximately 0.65, while the total linear wear of the pair increases systematically, exceeding 120 μ m at the end. In vacuum conditions, in the initial phase of the test run, the COF value is maintained at a level of approximately 0.70, and then rises to approximately 0.80. In this case, however, the wear of the friction pair is over an order of magnitude smaller (15 μ m) compared to the pair operating in air. The tribological characteristics of the pair 100Cr6–Al₂O₃ operating in air are comparable with the characteristics of the material pair made of 100Cr6 steel operating in

a vacuum, even though the degree of wear in the latter case is slightly greater. The pair $100Cr6-Al_2O_3$ operating in a vacuum exhibits markedly different friction and wear characteristics. It can be stated that, after a very short period of running-in, the COF stays at the level of 0.2, with a very slight downward trend (0.8 in air), and the intensity of wear is close to zero.

Another test sample was a pair with a low-friction coating of titanium-doped molybdenum disulphide MoS_2/Ti (**Fig. 3**). The coating can be described as low-friction based on the friction characteristics of the studied pairs. In all four cases, after the initial running-in period, the COF value did not exceed 0.1 and fluctuated

within a narrow range over the entire friction distance. Moreover, the wear values of the pairs, though varied, did not differ from one another significantly (negative values of wear intensity recorded in the initial period of friction, particularly in the steel balls, were due to heating and an increase in ball diameter).



Fig. 3. Changes in the coefficient of friction and total linear wear in the pair of counter-samples of 100Cr6 steel and Al₂O₃ with a disc of 100Cr6 steel with applied MoS₂/Ti coating





Fig. 4. Changes in the coefficient of friction and total linear wear in the pair with a disc of 100Cr6 steel with applied TiN coating



The final tribological tests were carried out for the pairs in which 100Cr6 steel discs were coated with TiN anti-wear coating (Fig. 4). In these cases, varying tribological behaviours were also observed in different material pairs assessed in air and in a vacuum. Adopting TiN-100Cr6 operating in air as a basis of reference for these pairs, it is found that the values of the COF in other cases are almost twice as high. Referring to the wear characteristics, the conclusion is that the properties of the TiN-Al₂O₂ pair operating in a vacuum are almost identical to the characteristics of the basis of reference. Markedly lower (more than 4-fold) wear intensities were observed for the pair TiN-100Cr6 operating in a vacuum. In the pair TiN-Al₂O₂ operating in air, they were over 10 times lower. This behaviour is very interesting, as are the COF fluctuations observed in these pairs working in a vacuum.

Figures 5 and **6**, and **Tables 1** and **2**, show the results of mean wear intensity and mean COF determined for the studied material pairs. The graphs demonstrate the variability of these parameters depending on the operating environment.





Rys. 5. Średnie wartości intensywności zużywania badanych skojarzeń materiałowych

Table 1.Mean values of wear intensity in the studied pairs: A – air, V – vacuumTabela 1.Zestawienie średnich wartości intensywności zużywania badanych skojarzeń: A – powietrze, P – próżnia

	100Cr6-	100Cr6-	MoS ₂ /Ti-	MoS ₂ /Ti-	TiN–	TiN–
	100Cr6	Al ₂ O ₃	100Čr6	Al_2O_3	100Cr6	Al ₂ O ₃
А	0.057242	0.003518	0.000865	0.000770	0.052860	0.003125
	± 0.010238	± 0.000957	± 0.000403	± 0.000179	± 0.000580	± 0.000233
V	0.019577	0.000525	0.002066	0.000616	0.012093	0.057910
	± 0.002001	± 0.000134	± 0.000438	± 0.000236	± 0.002482	± 0.003083

The intensity of wear in the pair 100Cr6–100Cr6 was almost 3 times lower (**Fig. 5**), and, in the TiN– -100Cr6 pair, they were nearly 4 times lower in the vacuum than in air. The replacement of the countersample with an Al_2O_3 ball paired with a disc made of 100Cr6 steel was associated with an over 5-fold reduction in wear intensity in the vacuum. In contrast, the pair with the applied TiN coating reversed the situation completely: The intensity of wear of this pair in the vacuum increased nearly 18-fold in relation to the intensity of wear in air. The intensity of wear of the pair $MoS_2/Ti-100Cr6$ in the vacuum was more than twice as high as the intensity of wear in air. It can also be seen that the degree of wear in the pair $MoS_2/Ti-Al_2O_3$ was the same in a vacuum as in air.

When analysing the effect of the environment on the coefficient of friction, it is important to highlight a different behaviour of the steel disk paired with 100Cr6 and Al_2O_3 balls (**Fig. 6**). The COF in the pair 100Cr6–100Cr6 was about 1.5 times higher when the pair operated in a vacuum than in air, whereas in the pair $100Cr6-Al_2O_3$ the value of the coefficient in air grew over 3.5 times compared to the vacuum.



Fig. 6. Mean values of COF in the studied material pairs Rys. 6. Średnie wartości współczynnika tarcia badanych skojarzeń materiałowych

	100Cr6– 100Cr6	100Cr6– Al ₂ O ₃	MoS ₂ /Ti– 100Cr6	MoS ₂ /Ti– Al ₂ O ₃	TiN– 100Cr6	TiN- Al ₂ O ₃
А	0.476	0.747	0.074	0.084	0.458	0.732
	± 0.038	± 0.086	± 0.020	± 0.009	± 0.066	±0.073
V	0.746	0.219	0.076	0.763	0.819	0.718
	± 0.059	± 0.018	± 0.008	±0.011	± 0.050	± 0.020

 Table 2.
 Mean values of coefficient of friction in the studied pairs: A – air, V – vacuum

 Tabela 2.
 Zestawienie średnich wartości współczynników tarcia badanych skojarzeń: A – powietrze, P – próżnia

The pair consisting of a TiN-coated disc and a steel ball operating in air was characterized by lower COF than the same pair operating in a vacuum; however, the ratios were even greater than in the steel pair (100Cr6–-100Cr6). The replacement of the counter-sample with Al₂O₃ did not produce major differences in the mean values of COF recorded in the vacuum and in air.

The evaluation of wear of the studied elements also involved images of wear tracks on the ball and disc recorded with optical microscope and interferometric profilograph. The results of geometric surface structure (GSS) measurements of different paired elements are listed in **Tables 3**, **6**, and **7**.

Comparing the total wear of the pair 100Cr6– -100Cr6 (**Fig. 5**) with images of wear of the disc and ball (**Table 3**), it can be concluded that the steel ball had worn out more in air than in the vacuum, and the relationship is proportional to the determined wear intensity. Similar ratios were observed in the case of the ceramic ball; however, after vacuum tests, the wear tracks both on the disc and the ball are very small.

A significant difference was noted in this pair with regard to the depth of wear tracks on the steel disc operating in air, as compared to the disc working in a vacuum (**Table 3**). Such a considerable degree of wear observed on the steel disc operating in air may be due to the presence of wear products in the form of iron oxides which intensified the wear of the steel sample. No such products were formed on the disc operating in a vacuum. The above finding was confirmed by chemical analyses conducted in an EDX microanalyser. The results are shown in **Table 4** and **Table 5**.

The wear tracks and GSS of the test elements in the pair containing a MoS_2/Ti -coated disc are listed in **Table 6**. It appears that, regardless of the ambient atmosphere, the friction tracks on the elements of the pair are small. Very good lubricating properties and a low degree of wear associated with coatings based on molybdenum disulphide MoS_2 have been demonstrated in a number of studies carried out under similar conditions by other authors [**L. 8, 9, 10, 11**]. However, the relationships between the COF values in air ($\mu \approx 0.2$) and in a vacuum ($\mu \approx 0.05$) observed in the sliding friction of a ball made of 9Cr18 steel coated with MoS₂ on a disc made of the same steel reported in the study [L. 8] are reversed. Similar results as in [L. 8] were obtained in the study [L. 9] in a sliding contact of a ceramic ball (Si₃N₄) with MoS₂ coating on steel (ANSI 52100), and, in the study [L. 11], in a sliding contact of a stainless steel chrome ball (440C) with MoS₂ coating on a silicone substrate. In the latter publication, the values of the coefficient of friction were $\mu \approx 0.1$ and $\mu \approx 0.05$ in air and in the vacuum, respectively. The wear values obtained in a vacuum were also lower than in air.

In the studies of the pair $MOS_2/Ti-100Cr6$ conducted by the authors of the present article, the coefficients of friction in air and in a vacuum varied only slightly, and it can be concluded, within the margin of statistical error (**Table 2**), that the total intensity of wear of the elements in a vacuum was over twice as high as in air, unlike in the study results reported in [**L. 11**]. The findings seem contradict the practice, as it is widely known that molybdenum disulphide, the main component of the studied coating, is used as a solid lubricant in aerospace mechanisms. Consequently, the problem should be explored in more detail.

The results of GSS evaluation after the friction tests of the pair elements in which the disc was coated with the anti-wear coating TiN are summarized in Table 7. The presented images of friction tracks on the balls and discs are significantly larger and more distinct than in the pairs in which the samples were coated with a lowfriction coating. It should be noted that the wear tracks seen on the 100Cr6 steel ball operating in air and on the Al₂O₂ ball operating in a vacuum do not vary in size; however, their counterparts that are visible on the discs are different. The above findings are confirmed by the surface roughness profiles determined for the friction tracks. In the pair TiN-100Cr6 operating in air, the material from the ball was transferred to the surface of the disc (Table 8). The transfer of the steel ball material to the interacting disc is documented by chemical analysis performed with a scanning microscope coupled with an EDX microanalyser. This is evidenced by images of iron Fe and oxygen O identified in the friction traces on the sample (disc).

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Table 3.	Tabela 3.





Table 4. Chemical analysis of the friction surface of the selected pair 100Cr6–Al₂O₃ operating in air Tabela 4. Analiza chemiczna powierzchni tarcia wybranego skojarzenia 100Cr6–Al₂O, pracującego w powietrzu

Table 5.Chemical analysis of the friction surface of the selected pair 100Cr6–Al2O3 operating in a vacuumTabela 5.Analiza chemiczna powierzchni tarcia wybranego skojarzenia 100Cr6–Al2O3 pracującego w próżni



The profiles of the wear track in the pair consisting of a TiN disc and ceramic ball Al_2O_3 operating in a vacuum reveal distinct wear of the disc exceeding the thickness of the coating, which was equal to 1.7 μ m (**Table 7**). Also, the distributions of elements in the tracks resulting from the interaction both on the disc and the ball (**Table 9**) point to the mutual transfer of material between the sample and counter-sample. This is evidenced by the mutually complementary images of oxygen O, iron Fe, and aluminium Al in the track of ball friction; whereas, the transfer of the ball material (Al_2O_3) is demonstrated by the presence of aluminium Al and oxygen O traces on the disc. The abrasion of the coating is clearly demonstrated by black streaks on the images of nitrogen N and titanium Ti (TiN coating) and the exposed path of iron Fe (substrate 100Cr6).

The graphs in **Fig. 5** show that the intensity of wear in the pair TiN–100Cr6 operating in air is comparable to the intensity of wear noted in the pair TiN–Al₂O₃ operating in a vacuum. This is confirmed by the images of ball wear tracks presented in **Table 7**.

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Table 6.	Tabela 6.



Summary of GSS of wear tracks on the disc (TiN) and ball depending on the material and ambient atmosphere Zestawienie SGP śladów zużycia na tarczy (TiN) i kulce w zależności od materiału i atmosfery otaczającej Tabela 7. Table 7.



Table 8. Chemical analysis of the friction surface of the selected pair TiN-100Cr6 operating in air

Tabela 8. Analiza chemiczna powierzchni tarcia wybranego skojarzenia TiN-100Cr6 współpracującego w powietrzu



Table 9. Chemical analysis of the friction surface of the selected pair TiN–Al₂O₃ operating in a vacuum Tabela 9. Analiza chemiczna powierzchni tarcia wybranego skojarzenia TiN-Al₂O₃ współpracującego w próźni



CONCLUSIONS

The incorporation of thin-film technologies into machines and equipment may bring a number of benefits, provided that the technologies are properly selected to match the working conditions. To achieve that, the mechanisms of friction and wear affecting the elements of friction joints coated by them need to be thoroughly explored. Among multiple studies in this field, important insights can be derived from analysing friction under vacuum conditions, i.e. with pressure reduced to a level at which nearly all effects of the environment are eliminated, enabling friction of physically clean surfaces. This is important for evaluating the behaviour of material pairs under conditions of dry friction, e.g., after rupturing the oxide film, removing moisture, etc., but also in lubricated conditions, in particular, after breaking the lubricant film, which not only can cause the destruction of rubbing materials, but also favourably affect, for example, the restoration of the boundary layer.

The findings above point to the possibility of conducting tribological tests characterized by good repeatability in the macro-scale corresponding to the actual friction elements. Some of them corroborate friction mechanisms and their effects, e.g., oxidative wear, expected and verified by other authors. In others, weakly correlating friction and wear characteristics in air and in a vacuum require further elucidation. For example, in the pairs of 100Cr6 and Al₂O₂, a surprisingly high coefficient of friction in air is accompanied by low wear intensity (with an interesting technical application revealing itself), unlike in the case of friction under vacuum conditions, where the values of both parameters are low. The opposite friction interaction is associated with the TiN coating paired with 100Cr6 steel. Under vacuum conditions, high friction resistances are accompanied by low wear intensity (and vice versa in air). Good tribological properties were demonstrated in studies of the coating with molybdenum disulphide regardless of the conditions (type of environment) and the friction partner.

The article deals with the studies of material configurations that can be currently considered as typical. However, the results obtained for them show that their tribological characteristics should not be taken for granted. It is clear that tests conducted in air should not be used to infer the characteristics existing under vacuum conditions, which also entails operation in direct contact with exposed surfaces (physically clean, e.g., after running-in or after the breakage of the lubricant film and rupture of the surface layer). The incorporation of vacuum tests into such studies is a way to expand the existing body of knowledge on such pairs. Combined with necessary new insights into

the elementary processes/phenomena determining the mechanisms of friction and wear, such knowledge will enable the formulation of more precise, practicallyoriented rules.

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