The influence of TiB$_2$ coating on the friction parameters in sliding pairs under lubricated friction conditions

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Abstract. The aim of the present work is to determine the influence of TiB$_2$ coating on the friction parameters in sliding pairs under lubricated friction conditions. The TiB$_2$ coating deposited on modified surface layers of ring specimens made of 46Cr2 steel were matched under test conditions with counterparts made of CuPb30 bearing alloys. The tested sliding pairs were lubricated with 15W/40 mineral oil and 5W/40 synthetic engine oil. The lubrication of friction area with mineral oil causes the reduction of friction force and temperature in the friction area, whereas the lubrication with synthetic oil reduces the wear of the bearing and the changes in the geometrical structure of the cooperating friction pair elements. Lubrication of the friction area in the start-up phase of the friction pair by mineral oil causes faster stabilization of the friction conditions in the contact area than in case of lubrication of the friction pair by the synthetic oil.

1. Introduction

Design and modernization of the means of transport involves the issues of the reliability and durability of the construction. The present technology aims to create the elements working under maximum unit pressure, at high relative speeds of movable elements and high temperature of operation of kinematics systems. The realization of these processes is possible by the application of surface layers with suitable tribological properties, such as wear resistance, low friction coefficient, corrosion resistance and seize resistance. The development of production processes of surface layers and the knowledge about a formation of the selected parameters of these elements allows for the use of hard thin coating in a friction pair as an element affecting performance characteristics in kinematic pairs. The creation of TiB$_2$ hard thin coating is possible with the use of a variety of vapour deposition techniques, magnetron sputtering, ion beam sputtering, electron evaporation, pulse electrode surfacing, laser surface engineering, enhanced plasma, electrode position from molten salts [1,6,7]. Titanium diboride (TiB$_2$) has useful properties, because it can be used under severe operating conditions, whereas conventional tribo-metals exhibit performance difficulties at larger loads, higher speeds, and higher temperatures. The properties that make this material so beneficial are: its extremely high hardness, its high Young’s modulus, its high melting point, wear resistance, corrosion resistance and electrical conductivity [2,4,5,8,16]. TiB$_2$ is limited in its mainstream use because of its inherent brittle nature. As the demand for the fabrication of high hardness, low coefficient of friction coatings increase, the need arises for the quantification and characterisation of suitable coatings.

Physical vapour deposition (PVD) technique is commonly used for generation of TiB$_2$ coatings [6,9]. The TiB$_2$ films deposited using this technique have gained increasing attention due to their
mechanical and tribological properties [8]. However, a detailed investigation of the tribological properties of systems with TiB₂ as a sliding partner is largely lacking. Many results have been published on tribosystems where TiB₂ was acting to increase the wear resistance by improving fracture toughness [10-11, 15].

The aim of the present work is to investigate the tribological behaviour of the TiB₂ coating as observed in sliding applications with a bearing alloy. Thus, it is crucial to determine the influence of the TiB₂ coating modification of the sliding pair elements on the operating conditions and wear under lubrication conditions. The tests were carried out under limited lubrication with the use of engine oil in order to determine the impact of the lubricant on processes of friction and wear.

2. Experimental work

The aim of the study is to determine the influence of the technological TiB₂ coating on the friction parameters in sliding pairs under lubricated friction conditions. The research involves the determination of work parameters of friction pairs as a function of engine oil and changeable parameters of the load of friction pairs. In the research, the friction pairs were composed of ring samples and a counterpart made of bushing bearings (figure 1).

![Figure 1. Friction pair; 1-ring sample, 2 counterpart](image)

In the experiments, TiB₂ coating was deposited on ring specimens created from 46Cr2 steel (0.46% C, 0.5% Cr, 0.65% Mn) (figure 2). The obtained TiB₂ layer was then matched under test conditions with counterparts made of SAE-48 bearing alloy and the area of friction was lubricated with the engine oils: 15W/40 mineral oil and 5W/40 synthetic oil (Table 1).

![Figure 2. Microstructure of the coating TiB₂](image)

| Parameter                              | 5W/40 mineral oil | 15W/40 synthetic oil |
|----------------------------------------|-------------------|----------------------|
| Kinematic viscosity at 100°C           | 14.5 mm²/s        | 14.5 mm²/s           |
| Viscosity index                        | 178               | 133                  |
| HTHS dynamic viscosity at 150°C        | 3.6 mPa·s         | 3.8 mPa·s            |
| Pour Point                             | -42°C             | -36°C                |

Stand test was conducted on a T-05 block on ring tester. The tests were carried out following an algorithm, which included the initial running-in of the samples and the correct co-operation process at
the pre-determined load parameters. The running-in was conducted on a test site at a pressure of 5 MPa until a complete adhesion of the ring specimen and the counterpart was achieved. At starting phase, it was assumed that that the pair would be accelerated from a speed of 0 to 500 rpm in 30 s and the friction coefficient was measured as a function of pressure. The friction force, the temperature in the friction area and the wear of materials of the elements of friction pairs were tested under pre-determined conditions: the ring specimen rotational speed of 100 rpm and the unit pressure of 5, 10, 15 and 20 MPa.

3. The results of the research

The work of friction pairs during the start-up stage was characterized by high dynamics of tribological processes, as a consequence of external loads which had an important influence on lubrication conditions.

![Figure 3. Friction coefficient in the friction pairs lubricated by mineral (a) oil and synthetic oil (b)](image)

The change of the friction coefficient in the start-up phase of friction pairs showed a fast growth of friction resistance up to the point of achieving a maximum value and then a decrease of friction resistance was observed with the growth of velocity (figure 3). When used to lubricate the friction area the mineral oil was observed to have had important influence on the course of friction coefficient with reference to the registered friction coefficient in friction pairs lubricated with synthetic oil. In the friction pairs lubricated with mineral oil, two groups of friction pairs were observed. At low unit pressures from 5 to 10 MPa, a significant decrease of the friction coefficient was observed. It was below \(\mu=0.02\) and at the velocity \(~0.8\) m/s the friction coefficient stabilized itself (figure 3a). In case of friction pairs under pressure of 15-20 MPa, the friction coefficient was higher and for these pairs friction coefficient was observed on the level of about \(\mu=0.06\) but already at the velocity \(~0.35\) m/s, the friction coefficient began stabilizing itself. In the friction pairs lubricated with the synthetic oil, the scattering of the friction coefficient from \(\mu=0.01\) to \(\mu=0.055\) was observed. The lowest friction coefficient was recorded at pressure of 5 MPa, the maximum friction coefficient occurred at pressure of 20 MPa (figure 3b). The stabilization of the friction coefficient in friction pairs under pressure of 5 MPa was observed at the velocity \(~0.25\) m/s whereas in friction pairs under pressure of 10 to 20 MPa, it was observed at the velocity \(~0.75\) m/s.

Another significant aspect in describing friction pairs is the determination of the value of start-up moment. The results of the measurements of the maximum start-up moment differed significantly. The use of the synthetic oil in the lubrication of the friction pairs generated a lower start-up moment in pairs working under pressure from 5 to 10 MPa (figure 4). The difference in the start-up moment between the studied pairs was \(~22\%\) at pressure of 5MPa and \(24\%\) at pressure of 10MPa. At pressure from 15 to 20
MPa, the start-up moment was about 6-7% lower in the pairs lubricated by mineral oil than in the pairs lubricated by synthetic oil. The recorded maximum start-up moment can be the basis for the design of the friction pairs in terms of providing an adequate level of load at start-up time or creating adequate friction conditions. This allows one to determine the energy necessary to properly start the machine and allows for the design of friction pairs in terms of heat load. The differences between the start-up moment in the studied pairs are mainly the result of the impact of the surface layer in the friction elements, the lubricant and the intensity of the formation of boundary layers which reduce the frictional resistance [12,14].

![Figure 4. Influence of the type of engine oil on the start-up moment in function of the load of a friction pair](image)

The influence of engine oils on the friction conditions in the friction pairs with elements coating TiB₂ was tested under pre-determined conditions, at the ring specimen rotational speed of 100 rpm and at the jump unit pressure of 5, 10, 15 and 20 MPa. Analysing the run of the friction coefficient for both mineral and synthetic engine oil under study, it is possible to distinguish different run patterns of the changes in friction resistance during the test (Fig. 6). In friction pairs lubricated by mineral oil, a very

![Figure 5. Friction coefficient in the friction pairs with the CuPb30 alloy lubricated by mineral oil (a) and synthetic oil (b) vs time of test](image)
quick stabilization of the friction coefficient can be observed following the initial start-up phase, and the value of friction resistance depends on the pressure value of a pair under study. With a maximum pressure of 20 MPa, the friction coefficient stabilizes itself at $\mu=0.085$, and with the pressure of 5 MPa, it does not exceed $\mu=0.057$. In case of pressure 10 and 15 MPa, a slow decrease of friction coefficient could be observed as the test time was passing by. Other changes could also be noticed in the friction pairs lubricated by synthetic oil. Under the maximum pressure of 15 and 20 MPa, stabilization of the value of a friction coefficient occurred, but under pressure of 5 and 10 MPa, an intensive increase of the value of a friction coefficient took place as the test time was passing by.

The lubrication of the friction area by mineral oil generated lower values of friction force than in pairs lubricated by synthetic oil (figure 6). These changes were particularly important at lower unit pressures. An increase of the unit pressure caused a decrease of the percentage difference and when the unit pressures accounted for 20 MPa, the difference between lubricated pairs was about 7%. The measurements of temperature in the friction area showed similar temperatures in friction pairs under pressure from 5 to 15 MPa. The lubrication by the synthetic oil generated higher temperature in friction pairs at the unit pressure of 20 MPa. Under these conditions of load of the friction pairs, the percentage difference between the friction pairs lubricated by mineral oil and synthetic oil was about 4%.

![Figure 6. Influence of the type of engine oil on friction forces and temperature depending on unit pressure (at 100 rpm and after 500 s)](image-url)
Figure 7. Wear of bearing alloy material vs. type of engine oil used for lubrication of friction pairs

In the friction contact area of the cooperating surface layers, the removal processes of the material occurred, caused by a relative movement of the friction elements. The results of the stand test have shown no measurable weight loss of the material of ring samples. Significant changes were observed only in the topography of the surface layer (Table 3). The application of the engine oils to the friction pairs has exerted an important influence on the wear of the bearing alloy - the use of synthetic oil reduced the wear intensity of the studied bearing alloy CuPb30 (figure 7). In conditions of lubrication by synthetic oil, the wear intensity of the CuPb30 bearing alloy was between 10 and 30% lower than that measured under lubrication by the mineral oil. On the other hand, under pressure of 5 MPa, lubrication by the synthetic oil generated higher wear than under lubrication by mineral oil, the difference in the value of wear was ~ 16%.

Table 2. Surface roughness of ring samples and counterparts before the test [μm]

| Parameter of surface roughness | Ring sample with TiB₂ coating | Counterpart CuPb30 |
|-------------------------------|-----------------------------|--------------------|
| Ra                            | 0.51                        | 0.48               |
| Rz                            | 4.4                         | 3.0                |
| Ry                            | 5.8                         | 3.4                |
| Sm                            | 41                          | 48                 |

The measurements of surface roughness of the friction pair elements after the tests revealed significant changes in the measurement parameters of roughness, as compared to the values of those parameters before the tests (Table 2). The measurements of the Ra parameter of ring sample showed that in the friction pair lubricated by the synthetic oil, the Ra parameter was higher than in the friction pairs lubricated by the mineral oil (Table 3). An important change of Ra parameter was observed in the friction pairs lubricated by the mineral oil - the Ra parameter decreased by 25% compared to the surface roughness before the tests, and when the friction pair was lubricated by the synthetic oil the Ra parameter decreased only by 6%. Similar changes of a surface layer were observed when the Ry and Ry parameters were measured. In case of Rz and Ry parameters, a greater decrease was observed in friction pairs lubricated by the synthetic oil. Measurements of the Sm parameter have shown its growth in case of lubrication by mineral oil by about 5%, and in case of synthetic oil – by about 10%.
Table 3. Surface roughness of ring sample and counterpart after test

| Parameter of surface roughness | Mineral oil | Synthetic oil |
|-------------------------------|-------------|---------------|
| Value [μm]                   | Change [%]  | Value [μm]    | Change [%]  |
| Ra                           | 0,38        | 0,48          |
| Rz                           | 3,2         | 3,1           |
| Ry                           | 4,6         | 3,7           |
| Sm                           | 43          | 43            |
| Counterpart                  |             |               |
| Ra                           | 0,60        | 0,48          |
| Rz                           | 4,9         | 4,2           |
| Ry                           | 8,9         | 7,6           |
| Sm                           | 121         | 105           |

The measurements of counterparts made of bearing alloys showed an increase of their surface roughness (Table 3). The surface layer measurements of the counterparts indicated more substantial changes than in case of ring samples, the largest of them exceeding even several dozen percent. The biggest changes were recorded for the parameter Ry, which in case of the mineral oil increased by 162%, and in conditions of lubrication by synthetic oil - by 124%. Also, significant changes were recorded for Sm parameters. The lowest change of surface roughness was observed in case of the Rz and Ra parameters. The measurements of the Ra parameter of counterparts have shown its growth in case of lubrication by mineral oil by about 20%, and in case of synthetic oil, a decrease of the surface roughness by about 4%.

4. Discussion

The start-up phase of sliding pairs indicates the behaviour of the system during its further work until the modified layer is used up. The most favourable operating conditions are presented in sliding pairs in which the friction coefficient increases in the initial stage of start-up, and then decreases significantly and stabilizes itself at a constant level. Those sliding pairs, which exhibited the tribochemical equilibrium within the shortest time generate optimal conditions for their further operation. The registered change of the friction force reveals the ability of the sliding pairs to adapt to the friction conditions in the extension of the pairs’ operation time. The stabilization of friction resistance indicates the adaptation of the pair to the existing forces and the generation of stable anti-wear and anti-seizure layers. The created surface layers ensure a separation of the co-operating elements of friction pairs and a reduced rate of direct adhesion between the surface irregularities. These conditions create a state of equilibrium between the processes of layer destruction and creation within the tribochemical processes occurring in the friction pair [3,14].

The differences in the wear of the bearing alloy and the absence of measurable wear of the surface layer ring specimen were the effect of the interaction between the cooperating surface layers, as well as physiochemical changes of their surfaces that were induced by external forces. The lubrication factor is crucial for these processes, because it creates favorable or unfavorable friction conditions depending on its transformation. These changes contribute to the generation of boundary layers on the surface layers of the cooperating elements that are either highly resistant to ruptures or are quickly destroyed under variable operating conditions [14]. These changes may lead to smoothening the surface and the removal of its irregularities, which eliminate the potential sources of damage of surface layer and stabilize the wear process. However, the hard wear products created in the friction process induce chipping, slicing, and grinding, which can intensify wear processes [13].
The surface roughness parameters measured, indicated the intensity of friction and its influence on the shaping of the sliding pairs' geometric structure. As an effect of the processes occurring within the friction area under the external forces, the system processes the preexisting geometric structures of both elements into a system with a structure that ensures the most favorable friction conditions [3]. As a result of these changes, a structure was created that reflected them, ensuring the given association of optimal functionality; that is, an operating surface layer was generated [12].

5. Conclusions
The following conclusions may be drawn on the basis of the experimental test performed and the analysis of their results:
1. Lubrication of the friction area by mineral oil reduces start-up moment, friction force and temperature in the friction area especially at higher unit pressure, whereas the lubrication by mineral oil reduces the wear of tested bearing alloy.
2. Lubrication of the friction area in the start-up phase of the friction pair by mineral oil causes a faster stabilization of the friction conditions in the contact area than when synthetic oil is used for lubrication of the friction pair.
3. The used synthetic oil in the friction pairs causes the reduction of wear of the CuPb30 bearing alloy by about 10 to 30% as compared to the friction pairs lubricated by mineral oil.
4. The surface roughness of the CuPb30 bearing alloy counterparts showed less change in the geometric structure of the friction pair elements lubricated by synthetic oil than lubricated by mineral oil.

Reference
[1] Berger M, Hogmark S 2002 Tribological properties of selected PVD coatings when slid against ductile materials, Wear 252 557–565
[2] Yuan J, Zhang Z, Yang M, Guo F, Men X, Liu W 2017 TiB2 reinforced hybrid-fabric composites with enhanced thermal and mechanical properties for high-temperature tribological applications, Tribology International 115 8–17
[3] Szczypinski-Sala W, ·Lubas J 2016 The use of surface layer with boron in friction pairs lubricated by engine oils IOP Conf. Series: Materials Science and Engineering, 148 1-9
[4] Lofaj F, Moskalewicz T, Cempura G, Mikula M, Dusza J, Filemonowicz AC 2013 Nanohardness and tribological properties of nc-TiB2 coatings, Journal of the European Ceramic Society 33 2347–2353
[5] Mikula M, Grančič B, Buršiková V, Csuba A, Držík M, Kavecký Š, Pleceník A, Kúš P 2007 Mechanical properties of superhard TiB2 coatings prepared by DC magnetron sputtering, Vacuum 82 278-281
[6] Berger M, Coronel E, Olsson E 2004 Microstructure of d.c. magnetron sputter TiB2 coatings, Surf. Coatings Technol. 185 240
[7] Sanchez CMT Plata B R, Maia da Costa MEH, Freire FL Jr. 2011 Titanium diboride thin films produced by dc-magnetron sputtering: Structural and mechanical properties, Surf. Coatings Technol. 205 3698–3702
[8] Wäsche R, Klaffke D, Troczyński T 2004 Tribological performance of SiC and TiB2 against SiC and Al2O3 at low sliding speeds, Wear 256 695–704
[9] Zhang TF, Gan B, Park SM, Wang QM, Kim KH 2014 Influence of negative bias voltage and deposition temperature on microstructure and properties of superhard TiB2 coatings deposited by high power impulse magnetron sputtering, Surf. Coat. Technol. 253 115-122
[10] Panish N, Wangyao P, Hannongbua S, Sricharoenchai P, Sun Y 2006 Tribological study of nano-multilayer ultra-hard coatings based on TiB$_2$, Rev. Adv. Mater Sci, 13, 117-124

[11] Nehme G, Mourhatch R, Aswath PB 2010 Effect of contact load and lubricant volume on the properties of tribofilms formed under boundary lubrication in a fully formulated oil under extreme load conditions, Wear 268 1129-1147

[12] Guha D, Chowdhuri R 1996 The effect of surface roughness on the temperature at the contact between sliding bodies, Wear 197 63-73

[13] Schouwenaars R, Jacobo VH, Ortiz A 2007 Microstructure aspect of wear in soft tribological alloys, Wear 263 727-735

[14] Nehmeb G, Mourhatcha R, Aswatha PB 2010 Effect of contact load and lubricant volume on the properties of tribofilms formed under boundary lubrication in a fully formulated oil under extreme load conditions, Wear 268 1129-1147

[15] Berger M, Hogmark S 2002 Evaluation of TiB$_2$ coatings in sliding contact against aluminium, Surf. Coat. Technol. 149 14-20

[16] Panich N, Wangyao P, Hannongbau S, Sricharoenchai P, Sun Y 2006 Multilayered systems of nanostructured TiB$_2$ thin films and its mechanical properties, Journal of Metals, Materials and Minerals. 16 45-50