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TRIBOLOGICAL PROPERTIES OF THE FRICTION PAIR WITH a-C:H COATING UNDER THE CONDITIONS OF LUBRICATION WITH 10W40 BIODEGRADABLE ENGINE OIL

WŁAŚCIWOŚCI TRIBOLOGICZNE PARY CIERNEJ Z POWŁOKĄ a-C:H W WARUNKACH SMAROWANIA BIODEGRADOWALNYM OLEJEM SILNIKOWYM 10W40

Key words: coating, wear, friction, surface layer, DLC, engine oil.

Abstract: The present study discusses the influence of an a-C:H coating on the tribological parameters of sliding pairs under mixed friction. Using the PVD method, the a-C:H coating was formed on specimens made from AISI 4337 steel. The sliding friction and wear process was carried out on the pairs of AISI 4337 steel and SAE-48 bearing alloy, which were lubricated with 10W40 biodegradable engine oil. The investigation showed significant differences in the friction coefficient and temperature in the tested pairs with the steel surface layer and the a-C:H coating. In the friction pairs with the a-C:H coating, the tested parameters of friction were higher than in pairs with the steel surface layer. The pairs with the a-C:H coating showed more intensive wear of the SAE-48 bearing alloy than those with a steel surface. The surface layer used in a friction pair leads to the deterioration of the lubricating properties of engine oil and reduces its resistance to scuffing.

Słowa kluczowe: powłoka, zużycie, tarcie, warstwa powierzchniowa, DLC, oleje silnikowe.

Streszczenie: W pracy przedstawiono wpływ powłoki a-C:H na parametry tribologiczne par ciernych w warunkach tarcia mieszanego. Powłoka a-C:H została wytworzona na próbkach wykonanych ze stali AISI 4337 metodą PVD. Proces tarcia ślizgowego i zużycia realizowano w skojarzeniu stal AISI 4337 i stop łożyskowy SAE-48 smarowanych biodegradowalnym olejem silnikowym 10W40. Badania wykazały istotne różnice w wartości współczynnika tarcia i temperatury w badanych parach ze stalową warstwą wierzchnią i powłoką a-C:H. W parach z powłoką a-C:H rejestrowane parametry tarcia były wyższe niż w parach ze stalową warstwą wierzchnią. Pary z powłoką a-C:H wykazywały intensywniejsze zużycie stopu łożyskowego SAE-48 niż pary z powierzchnią stalową. Warstwa wierzchnia zastosowana w parze ciernej prowadzi do pogorszenia właściwości smarnych oleju silnikowego i zmniejsza jego odporność na zacieranie.

INTRODUCTION

The exploitation of friction pairs under conditions of variable tribological extortions causes unfavourable changes in the structure of materials, which may cause the loss of essential functional properties and, in extreme situations, even lead to the destruction of the cooperating structural elements. Significant tribological properties usually do not depend on the possibility of transferring mechanical loads through the entire cross-section of the element but mainly on the structure and properties of the surface layers. The continuous increase in the requirements for construction materials influences the production of new types or modifications of the existing anti-wear and anti-seize solutions, e.g., by changing the chemical composition of the coating or the technology of its production. To properly

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protect the surface and reduce the wear processes, the top layers and coatings are expected to have a combination of properties such as high hardness, resistance to shear, bending, cracking and good adhesion to the substrate.

Carbon coatings are widely described and tested and characterised by favourable performance properties. From a tribological point of view, the most important of them is high hardness, low friction coefficient (≤0.2 against steel under lubricant free), low thermal expansion coefficient and wear resistance in conditions of dry friction or limited lubrication **[L. 1, 2, 14, 15]**. An important advantage of the carbon coating (a-C:H) is also the process of its production, where the temperature of the deposition process does not exceed 250°C, which allows for maintaining the hardness of the substrate on which the coating is made **[L. 9, 10]**.

Some authors point to a significant problem with selecting a lubricant for hard low-friction coatings, as they are currently used mainly for steel friction nodes. Using these lubricants in pairs with carbon coatings may be harmful under certain friction conditions, resulting from different conditions in which the lubricating film is formed **[L. 11]**. DLC coating studies by Mustafa et al. under boundary lubrication conditions showed a significant influence of the type of lubricant, but also of oil additives, on the tribological properties. With appropriate additives to base oils, DLC coatings ensure low friction coefficient values and high wear resistance **[L. 16, 17]**. Tests of DLC coatings carried out in conditions of dry friction and lubrication with Ringer's fluid in the ball-plate contact showed lower wear and lower friction coefficient in pairs with the a-C:H coating than in pairs with steel samples. Rapid jumps of the acoustic emission signal were also observed in these friction pairs, which were related to the cracking of the coating during the friction process under load conditions **[L. 14]**. High-hardness materials usually have lower fracture toughness, which causes damage to the coating due to microcracks, and in the case of the DLC coating, the wear is accelerated by a fracture across the entire thickness of the coating, which significantly increases the wear compared to abrasive wear **[L. 18]**. The research also showed that the coefficient of friction for the a-C:H coatings decreased due to the graphitisation of the coating, which also increases the wear rates **[L. 18]**. Under these friction conditions, the coating undergoes wear processes as a result of the combined action of abrasive wear and tribochemical wear, which affects the destruction processes of the cooperating elements of the friction pair **[L. 6]**. There was also an adverse effect of the temperature increase in the contact area on using these coatings in friction pairs **[L. 13, 15]**. This is particularly important in conditions of limited lubrication, where the peaks of the surface roughness of the cooperating surface layers come into contact with each other.

The study aims to investigate the influence of the a-C:H coating on friction and wear processes in friction pairs lubricated with 10W40 biodegradable engine oil. This paper also describes the anti-seizure parameters of the engine oils used for lubricating the sliding pairs.

MATERIALS AND METHODS

AISI 4337 steel is a heat treatable, low alloy steel generally used in mechanical engineering, including the construction of heavily loaded elements in internal combustion engines (**Table 1**). Steel ring specimens were heat-treated (38 HRC ±2), and the final surface treatment was grinding (**Fig. 1**). As an anti-wear coating, a commercial a-C:H coating was used, produced by the PVD method, and it was formed according to the technological parameters used by the manufacturer. The coating was applied to the steel surface (coating thickness 2 μm, coating hardness 10–15 GPa). It was assumed that the coating properties, i.e., hardness and thickness, were within the manufacturer's specifications. The counterpart with an SAE-48 alloy was cut from a journal bearing (**Table 1**) with dimensions of 15.75 x 6.35 mm (**Fig. 1**).

Table 1. Chemical composition of specimens (wt.%)
Tabela 1. Skład chemiczny próbek pierścieniowych

Material	C	Cr	Mn	Mo	Ni	Si	S and P	Fe	Pb	Cu
AISI 4337	0.34	1.5	0.65	0.23	1.5	<0.4	<0.035	Balance	–	–
SAE-48	–	–	–	–	–	–	–	–	26–33	Balance

Comparative tribological studies were carried out in conformal contact with the lubrication of the friction area with the 10W40 biodegradable engine oil, fully synthetic, environment-considerate low SAPS oil (low Sulphate Ash, Phosphor and Sulphur content) for modern diesel aggregates. The aforementioned oil is particularly suitable for diesel engines with exhaust treatment systems (**Table 2**).

Table 2. Characteristics of engine oil 10W/40
Tabela 2. Charakterystyka oleju silnikowego 10W/40

Parameter	Value
Kinematic viscosity at 40°C	91.1 mm²/s
Kinematic viscosity at 100°C	14.3 mm²/s
Viscosity index	163
Biodegradability acc. to	> 60%
OECD 301 B	ACEA E6, E7, E9
Specification	API CJ-4/SN
	CAT ECF-3, ECF-2, ECF-1-a

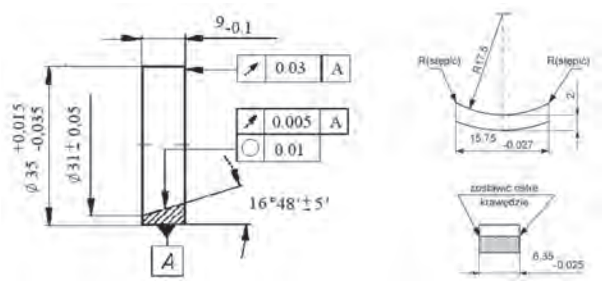

Fig. 1. Dimensions of elements of the tested pair: 1 – ring specimen, 2 – counterpart
Rys. 1. Wymiary elementów pary ciernej: 1 – próbka pierścieniowa, 2 – przeciwpróbka

The test included the measurements of the friction pair cooperation under unit pressure of 5, 10, 15 and 20 MPa. The ring specimen during the test had a rotational speed of 100 rpm, and the friction test continued for 1000 s. The measured parameters during the friction test were friction coefficient, the temperature in the friction area and mass wear. The tests were conducted on the ring tester's T-05 block, and the engine oil lubricated the ring specimen.

The roughness of the friction pair elements was measured using a Taylor Hobson Surtronic 3+ roughness meter. The measurements were made on the working surfaces of the elements, perpendicular to the ring movement direction. The measurements were conducted in accordance with ISO 4287 standards.

The scuffing test of engine oil in the sliding movement was performed utilising the four-ball testing machine. The balls with a diameter of 12.7 mm (0.5 in.) were used in the tests. The ball's surface roughness, expressed as the Ra parameter, equalled 0.032 µm, and hardness amounted to 62 HRC ± 2. All the tests in the present research were repeated three times.

RESULTS

The after test measurements of surface roughness of the elements of friction pairs (ring specimens and counterparts) revealed significant changes as compared to the roughness prior to the tests. The Ra and Rz parameters of surface roughness measurements indicated a decrease in surface roughness. The Ra parameter measurements showed a change of 27% of the surface layer of steel ring specimens and 10% of the surface layer of the a-C:H coating (**Table 3**). The registered values for the Rz parameter showed a decrease of 19% of steel ring specimens and 32% of ring specimens with the a-C:H coating. However, measuring the RSm parameter indicated an increase of its value by 14% on the steel surface layer and stabilisation on the surface layer with the a-C:H coating.

Table 3. Roughness of ring specimens after test (20 MPa load)
Tabela 3. Chropowatość próbek pierścieniowych po teście (obciążenie 20MPa)

	Steel		**a-C:H coating**	
Parameter	Value [µm]	Change [%]	Value [µm]	Change [%]
Ra	0.164	-27	0.232	-10
Rz DIN	1.63	-19	1.72	-32
RSm	34.4	14	32.2	-0.3

Table 4. Roughness of counterparts after test (20 MPa load)
Tabela 4. Chropowatość przeciwpróbek po teście (obciążenie 20MPa)

	Steel		a-C:H coating	
Parameter	Value [µm]	Change [%]	Value [µm]	Change [%]
Ra	0.284	-33	0.317	-25
Rz DIN	3.21	-31	4.47	-3
RSm	100.1	48	66.5	-2

The surface roughness of the counterparts from SAE-48 bearing alloy showed significant decreases in the measured parameters (**Table 4**). The smallest changes were observed for the Rz parameter, which decreased by 31% on the surface layer of the counterpart after cooperation with the steel surface layer and only 3% after cooperation with the a-C:H coating. Measurements of the Ra parameter showed a greater change in surface roughness: 33% and 25%. Particularly significant changes relate to the RSm parameters, which increased by 48% after the test with the steel ring specimens and decreased by 2% with the a-C:H coating.

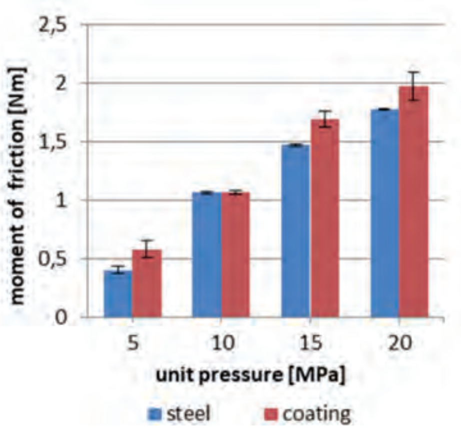

Fig. 2. Moment of friction during start-up of the pair
Rys. 2. Moment tarcia podczas rozruchu pary ciernej

The maximum friction moment is a significant parameter measured in kinematic pairs during start-up. The friction moment in the tested friction pairs was lower in pairs with steel ring specimens. Only during start-up, under the load of 10 MPa, the measured value was similar for the pairs with steel ring specimens and the one with a coating. An important difference in the start-up moment is observed at the load of 5 MPa, which is then 45% higher in the pair with ring specimens with coating than in the pair with steel ring specimens. At the load of 15–20 MPa, the difference between the values of start-up moment decreases and amounts to 15 and 10% accordingly (**Fig. 2**).

a)

b)

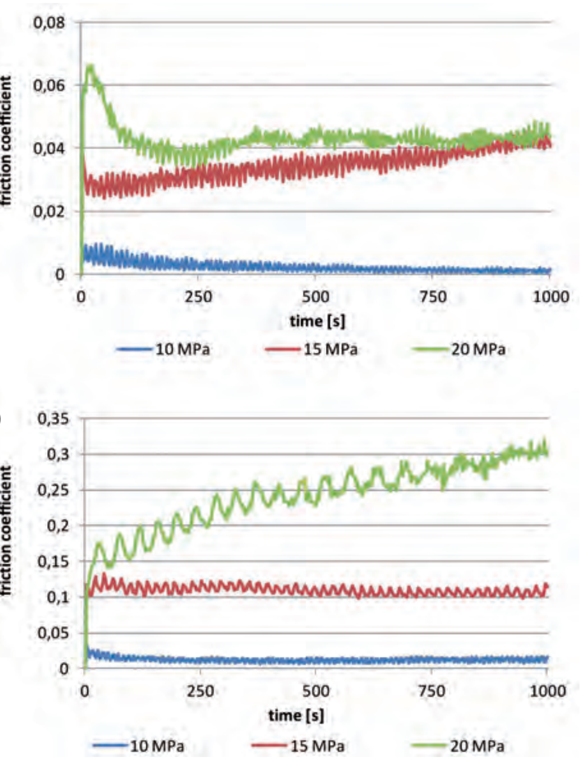

Fig. 3. Friction coefficient in fiction pairs with (a) steel ring specimens and (b) ring specimens with coating
Rys. 3. Współczynnik tarcia w parze ciernej z (a) stalowymi próbkami pierścieniowymi i (b) próbkami pierścieniowymi z powłoką

The friction resistance in the friction pairs with coating in the start-up period showed a higher friction coefficient than in pairs with a steel surface layer (**Fig. 3**). The courses of the friction coefficient in **Fig. 3** are selected from three test runs for each unit pressure value in the tested friction pairs. In pairs with a steel surface layer, for loads of 10-20 MPa, the friction coefficient increased at the moment of start-up of the friction pair, and the friction coefficient decreased further with the duration of the test. For the load of 20 MPa, the value of the friction coefficient stabilised after 200 s, and, at the end of the test, the friction coefficient value was about 0.04. A similar course of the friction coefficient was registered for the lowest load (5 MPa), stabilising at the value of about 0.002. At the load of 15 MPa, the friction coefficient slowly increased, and, at the end of the test, the value of the friction coefficient was about 0.04, too (**Fig. 3a**). In the pairs containing a surface layer with coating, the course of the friction coefficient was similar to that of the pair with steel surface layer, at the load of 10 MPa. However, at the end of the test, the friction coefficient stabilised at the level of 0.01, which was higher than in the pair with a steel surface layer. At the load of 15 MPa, the friction coefficient stabilised at over 0.1 right after start-up. The variable value of the friction coefficient at the load of 20 MPa led to its stabilisation at the level of 0.3 after less than 900 s (**Fig. 3b**).

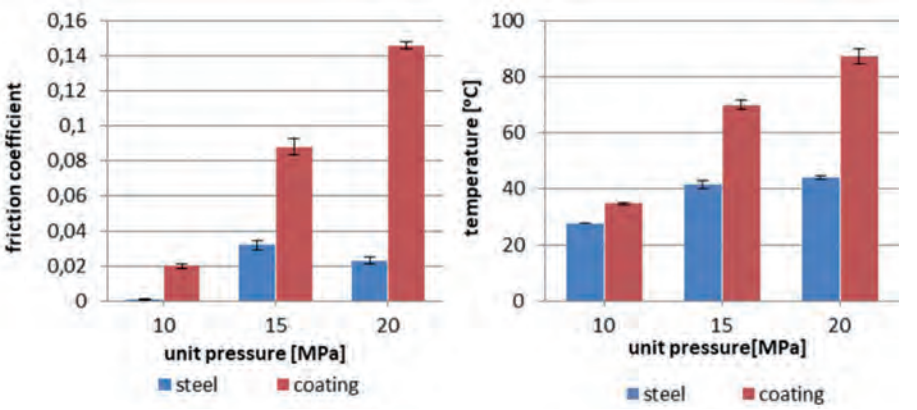

Fig. 4. Friction coefficient and temperature in friction area vs unit pressure
Rys. 4. Współczynnik tarcia i temperatura w obszarze tarcia w funkcji nacisków jednostkowych

The friction coefficient and the temperature at the end of the test allow for determining the friction pair's working conditions during further working conditions. Both parameters were higher in the friction pairs with coating than in the case of the pairs with steel ring specimens (**Fig. 4**). The greatest difference in the friction coefficient occurred at the load of 20 MPa, and it was six times lower in pairs with steel ring specimens, as compared to the pairs containing specimens with coating. At the load of 15 MPa, the friction coefficient was about 2.5 times lower for the steel ring compared to the coating ring. The measured temperature in the friction area also showed a very similar course to the friction coefficient of the tested friction pairs. At the load of 15 MPa, the temperature in the friction area was about 69% higher in pairs with a coating compared to the pairs with steel specimens, and, at the load of 20 MPa, it was about 98% higher. At 10 MPa, the temperature was about 25% higher in pairs with a coating than in pairs with steel specimens (**Fig. 4**).

The wear analysis of the friction pair elements did not show the measurable linear wear of the ring specimens, but it was possible to observe the intense wear process of the SAE-48 bearing alloy. The wear of the SAE-48 bearing alloy in the friction pair with coating was higher than in the friction with steel specimens (**Fig. 5**). The wear of the bearing alloys increased with the rise of pressure in the contact area between surface layers of both elements of

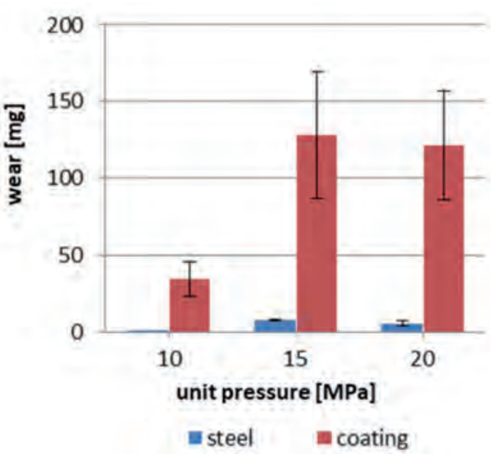

Fig. 5. Mass wear of SAE-48 bearing alloy
Rys. 5. Zużycie wagowe stopu łożyskowego SAE-48

the friction pairs. The difference in mass wear of the bearing alloy was very significant, and the difference between the alloy wear of the pair with a coating was about 20 times higher than in the pair with steel specimen.

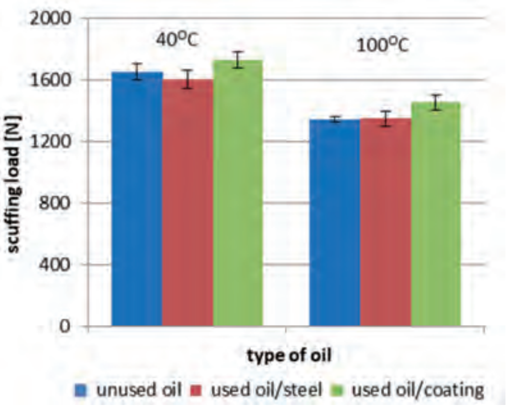

Fig. 6. Scuffing load of the unused and used oil
Rys. 6. Obciążenie zatarcia olejów nowych i zużytych

Measurements of the scuffing load of the used and unused 10W40 biodegradable engine oils showed high seizure resistance of the oil used in pairs with a coating during the wear tests of the friction pair, as compared to the unused oil (**Fig. 6**). In the tests carried out at the temperature of 40 and 100°C, it could be observed that used oils in pairs with a coating showed the highest scuffing load. At the temperature of 40°C, this difference accounted for 5%, compared to the new oil, while at the temperature of 100°C, it was higher and amounted to 8%. Used oil in pairs with steel specimens showed the lowest resistance to scuffing at the temperature of 40°C, and at the temperature of 100°C unused oil had the lowest seizure resistance. Also, at the temperature of 100°C, unused oil and used oil in pairs with steel specimens had a similar value of the scuffing load. A significant reduction of the scuffing load was observed at a higher temperature (100°C), and for the new 10W40 biodegradable oil, it decreased by about 19% and about 16% both for used oil in pairs with steel specimens and for used oil in pairs with a coating.

The pressure of seizure calculated for the tested oils showed significant differences, and it can be concluded that the higher temperature of the test showed a reduction in the pressure values, especially for used oil used in the pair with steel specimen (**Fig. 7**). At the temperature of 40°C, a higher pressure of seizure value is noticeable for the used oil in the pair with a coating (6%), as

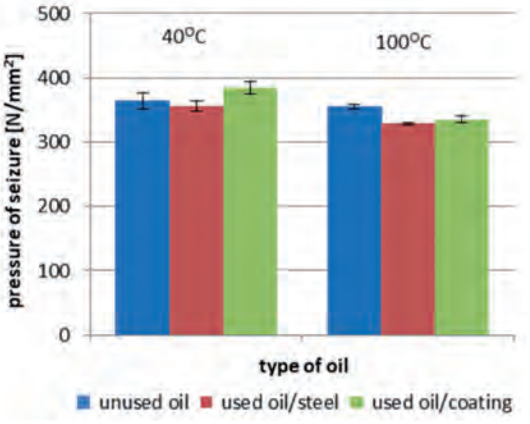

Fig. 7. Pressure of seizure of the unused and used oil
Rys. 7. Nacisk zatarcia olejów nowych i zużytych

compared to the unused oil. In the case of used oil in the pair with a steel specimen, the pressure of seizure value was lower by about 2% compared to the unused oil. At the temperature of 100°C, the pressure of seizure decrease was measured for the used oil in both tested friction pairs, and in relation to the unused oil, this decrease was 8% for the used oil in the pair with steel specimens, and 6%, respectively, for the used oils in pairs with a coating. The pressure of seizure showed a deterioration in the performance properties of the tested engine oils, which may have been caused by contamination of the oil with the wear products and a reduction in the amount of anti-seize additive in the oil. In friction conditions with limited lubrication in the friction area, the anti-seize additive contained in the engine oil is responsible for forming boundary layers preventing seizure, which leads to its wear. On the other hand, increases in the scuffing load can be explained by the penetration of coal, which exhibits lubricating properties.

DISCUSSION

Lubrication of the friction pairs with steel specimens and specimens with coating and structural elements by 10W40 biodegradable engine oils significantly shapes the processes of friction and wear by the formation of boundary layers as a result of the reaction of the surface layers of these elements and the lubricant. The observation of the geometric structure of the surface layers shows a significant reorganisation, the effect of which is an increase in the surface roughness of both layers, especially the surface of counterparts of the SAE-48 bearing alloy **[L. 3]**. Changes in the surface roughness parameters show a comprehensive picture of the surface, and the changes in the surface roughness result mainly from the adaptation of both surface layers of the cooperating elements to the specific load conditions of the friction pair. The changes in this parameter significantly affect the load-bearing capacity of the operating surface layers formed in the process of friction **[L. 4]**. An observation of the course of the friction coefficient shows that low unit pressures influence the shaping of favourable lubrication conditions, and the system changes the existing geometric structure of both elements into a composition, ensuring the most conducive cooperation conditions. The resulting structure provides the given pair with optimal functionality, caused by the creation of stable operational surface layers on both elements of the friction pair **[L. 5, 6]**.

The friction and wear processes taking place in the contact area of the sliding pairs depend on unit pressure between cooperating surface layers, microstructural changes of surface layer, chemical reactions between materials, material transformation, and material properties variation in the surface layers **[L. 3, 7]**. The wear of the bearing alloy is mainly caused by the hard areas in the surface layer of the second material and hard wear particles, which leads to the interaction between the two surface layers and a more intense abrasion of the softer material **[L. 5, 8]**. The hard wear products created in the friction process induce chipping, slicing, and grinding, which intensify the wear process **[L. 6]**.

During the operation, engine oil changes its properties and accumulates impurities, which deteriorate the lubrication conditions and increase the intensity of wear processes. The lubrication process can remove wear products from the friction area. However, due to tribochemical reactions, corrosion processes, cavitation wear, or fatigue wear may occur, the latter caused by dynamic changes in the pressure in the friction area. In addition, in the case of these coatings, a reduction in the surface fatigue strength is observed, which may intensify the wear processes of the friction pair **[L. 12]**.

CONCLUSIONS

Based on the experimental test, the following conclusions can be drawn:

1. The surface roughness of the SAE-48 bearing alloy and ring specimen with the a-C:H coating shows tendencies to decrease, and only the RSm parameter increases on the surface layer of the steel ring specimen.
2. The friction coefficient in the pair with the a-C:H coating is considerably higher than in the pair with steel specimens, and the temperature in the friction area is also higher, especially during the cooperation of the friction pair under higher unit pressure.
3. The application of the specimens with the a-C:H coating in the friction pair causes a significant increase in wear of the SAE-48 bearing alloy, together with the increased unit load in the friction area.
4. The wear processes lead to the destruction of engine oil and the deterioration of their resistance to scuffing, especially during the operation under higher temperatures.

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