

Krzysztof DRUET^{*}, Jacek ŁUBIŃSKI^{*}, Antoni NEYMAN^{*}

WEAR OF Si OR B DOPED AMORPHOUS CARBON COATINGS

ZUŻYCIE AMORFICZNYCH POWŁOK WĘGLOWYCH DOMIESZKOWANYCH Si LUB B

Key words:

DLC coatings, wear testing, biomedical tribology, ball-on-flat, distributed contact

Słowa kluczowe:

powłoki DLC, badania zużycia, tribologia biomedyczna, kula-płaszczyzna, styk konforemny

Abstract

The paper describes a complex wear research on DLC (amorphous carbon) doped with boron (B) and silicone (Si). The tests were carried out in two different sliding systems: flat-on-flat (ring contact, unidirectional sliding) and ball-on-flat (reciprocating sliding). Water and medical saline solutions were used as lubricants. A comparative analysis of the results revealed differences in the initiation and progress of wear and the endurance with regard to coating's composition, lubricant and contact geometry. The results obtained from the tests show the influence of the additive element on the wear resistance and the high dependency of coatings' performance in the type of contact and of the lubricant used. In all cases analysed,

^{*} Gdańsk University of Technology, Faculty of Mechanical Engineering, ul. G. Narutowicza 11/12, 80-233 Gdańsk, Poland.

the use of medical saline solution resulted in accelerated wear or an increased result scatter in comparison to pure water lubrication.

INTRODUCTION

Over the last decades, there is an increasing interest in the use of hard coatings on conventional construction materials for mechanical design. The typical purpose of coating application is to increased resistance to wear inflicted by friction. The diamond like carbon (DLC) stands out as the most promising material. It is a form of amorphous carbon with a high content of sp³ bonds typical to diamond. The coating can be a 'pure' form of DLC composed almost entirely of sp³ bound carbon. Common variations of such coating are hydrogen content, graphite type sp² bonds, metals, and other added compounds. The coating is usually applied directly to the working surface of the specimen or onto an intermediate sublayer. It is known that silicone additive improves wear resistance, stability in aqueous environments, and at elevated temperatures. Additives of metals or metal carbides improve the coating's adhesion to metallic substrate, thermal stability, and increases hardness [L. 2, 3, 4]. The light elements **B** and **Si** are known to improve biocompatibility and thermal stability of the coating [L. 4]. In addition, the authors' own experiences confirm that the wear resistance of DLC type coatings can be improved by adding selected elements to the material [L. 19].

The paper presents the results of an investigation on the behaviour of carbon based hard coatings in sliding contacts lubricated with water or medical saline solution. The research was primarily meant to advance knowledge on coatings potentially applicable in medical implants to the benefit of durability and patients' comfort [L. 5]. Some coatings are potentially useable and beneficial in medical tools, especially as anti-wear treatment. A secondary target of the research was to compare results from two different tribological tests: in concentrated contact in reciprocating motion and distributed contact in unidirectional motion. The first configuration bears resemblance to human joint geometry, and the second is typical to certain components in mechanisms. Both types are used in tribological testing and conclusions are drawn from just one type, so two tests were run alongside and results were interpreted jointly. A stainless steel was selected as a specimen substrate material and for all the components in contact with the specimens and lubricants in order to minimise corrosion related effects, such as reduced adhesion caused by water and NaCl solution penetration through coatings' imperfections and wear marks.

DEPOSITION OF COATINGS

Two types of coatings were used: silicon doped, hydrogenated amorphous carbon (Si-C:H) and boron doped, hydrogenated amorphous carbon (B-C:H).



Table 1. Coating process parameters

Tabela 1. Parametry procesu nakładania powłok

Residual gas pressure	2e ⁻³ Pa
Working pressure	0.3–0.38 Pa
Argon process gas output	50 cm ³ /min
Arc discharge current	70 A
Target sputtering power (all targets)	1.5 kW
Acetylene process gas output	16–42 cm ³ /min
Substrate polarisation potential	–100– –800 V
Process temperature	~ 200°C
Rotational speed of the instrument table	3 RPM

Application of coatings was done by pulse–reactive magnetron sputtering [L. 6]. High purity metals were used as targets: Ti – 99.9%, and Cr – 99.8%. The reactive process gas was acetylene (96.8% pure) and the protective atmosphere gas was argon (99.99 % pure).

Coatings were deposited on substrates of stainless steel. Either on balls Ø10 mm of AISI 440-C grade steel or on flat faces of cylindrical or block specimens made of 3H13 grade steel acc. to PN standard, equivalent to X30Cr13 grade acc. to EN and DIN standards and 420S45 acc. to BS standard.

All the surfaces to be coated were polished to a roughness $R_a < 0.02 \mu\text{m}$ and cleaned to chemical purity in a multi-stage process ending in the vacuum chamber. The deposition was carried out in a vacuum of less than 2×10^{-3} Pa. The surfaces were primed with a $0.3 \mu\text{m}$ thick layer of chromium. Then a transitional layer of chromium and the additive (Si or B₄C) was applied by magnetron sputtering. Next, the acetylene process gas was introduced into the chamber. The process was continued until a layer of desired thickness was deposited. A summary of the parameters of the coating process is given in **Table 1**.

SCRATCH TESTING OF COATINGS' ADHESION

The prevalent bond in the carbon coating is of sp², only a small proportion of the bond belongs to the sp³ type and a trace content of sp¹ bonds. The resulting coatings were thus relatively 'soft' – hardness in the range of between 8 and 10 GPa. The coatings are nano-composites, thanks to dispersed nano- crystallite carbides in the amorphous structure of the carbon coating [L. 6]. Short technical characteristics of the three coatings are presented in **Table 2** for comparison.



Table 2. Hardness and adhesion characteristics of the coatings used in testing

Tabela 2. Twardość i charakterystyki adhezji powłok użytych podczas badań

Additive element	Additive content	Coating thickness*	Hardness**	Coatings' adhesion strength***		
				L_{C1} [N]	L_{C2} [N]	L_{C3} [N]
B	2	2.5 ± 0.1	11.4	8	15	41
B	5	2.6 ± 0.1	12.6	8	16	46
Si	5	2.9 ± 0.1	12.6	5	12	32

* Coating thickness was measured by optical microscope observation of the edge of coating after rotating ball grinding.

** Vickers hardness test was used in a system composed of a Nephot2 optical microscope and a Hannemann micro hardness tester. Test load: 0.5 N. Jönsson – Hogmark hardness correction.

*** A Revetest® [18] scratch tester was used to measure coatings' adhesion. Three critical loads were measured: L_{C1} – load to initiate cohesive damage to the coating (cracks), L_{C2} – load causing cracking across the scratch and splintering at the edge of the scratch, L_{C3} – load at which coating is removed at the bottom of the scratch.

WEAR TESTING METHOD – UNIDIRECTIONAL ROTATIONAL SLIDING

The tests in distributed (flat) contact were carried out with the use of a PT-3 tribometer [L. 17, 18, 20, 21]. The specimen arrangement adopted for the task is presented in Fig. 2. Both specimens are cylindrical in shape with radial grooves across the face on one end of the cylinder for improved lubricant access and wear debris evacuation. The total contact area is approx. 144 mm^2 divided into 4 identical crescent shaped segments (Fig 1). Immersion lubrication was used.

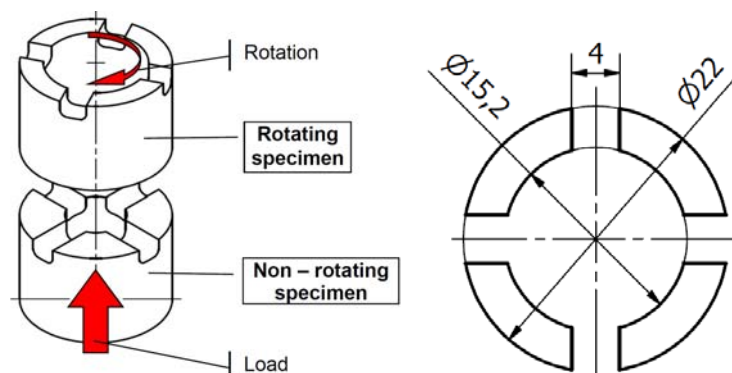


Fig. 1. The general arrangement of the sliding contact used in DLC coating testing (left) and footprint of the geometric contact area (right)

Rys. 1. Ogólny widok układu o tarczu ślizgowym stosowanego na tribometrze PT-3 w badaniach powłok DLC (z lewej) oraz rzut nominalnej powierzchni styku (z prawej)

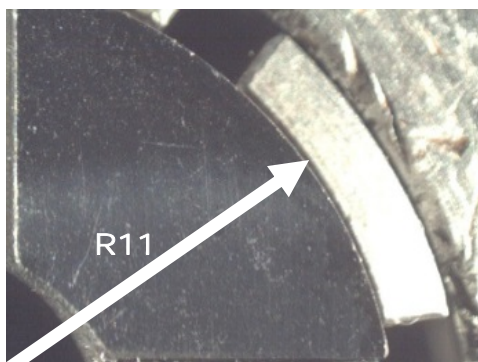


Fig. 2. Photography of unused lower specimen's face segment

Rys. 2. Widok segmentu dolnej próbki z powłoką DLC przed użyciem

The selected test conditions were a constant sliding velocity (at mean sliding radius) of 0.2 m/s ($-0.001/+0.007$) and a constant load given as an average contact pressure of 20 MPa. An intermittent test scheme was used with optical examination of specimens between test parts. Digital images of the lower specimen's face were taken at examination, initially every 3 minutes, with progressive run extension up to 60 minutes in a single run. The damage criterion was the removal of a portion of the coating sufficient for the stainless steel substrate to get into the sliding contact, which was in all cases tantamount to massive damage of the working face. Cracks and chipping were not considered as critical as long as there was no evidence of a comprehensive wear. The progress of wear can be described as typical, with a long period of slow progress followed by rapid damage to the coating, concluding the test cycle.

An image of a quarter of the lower specimen's unused face is presented in **Fig. 2**. The damage on a critical scale can be seen in similar images collected in **Fig. 3**, showing examples of portions of the specimens' faces after completed tests. A minimum of 3 repetitions were made for each combination of parameters.

In all three coatings, the most prominent difference between the tests carried out with pure water lubrication and with saline water solution is that the spread of results is much greater in the latter case. The proportion between the maximum and minimum endurance, when lubricated with saline solution, is 15.3 with DLC + Si 1% coating, 4.3 with DLC + B 2% coating and 16 with DLC + B 5% coating. If lubricated with water, these proportions are 1.5, 2.3, and 1.05, respectively. This result indicates to an existing influence of the presence of NaCl in the lubricating solution on the kinetics of wear under the test conditions considered. The average values of endurance indicate that the silicon-doped coating is superior to boron doped in test conditions in both

lubrication cases. All coatings have a much lower minimum endurance result when lubricated with saline solution.

In case of a distributed contact, as used in the experiment, there is a significant potential for secondary damage within the contact zone due to the action of wear products. With hard coatings, like DLC, the progress of the delamination of the top layer might be the factor controlling the endurance of the contact. It may be hypothesized that, if the wear particles are sufficiently large, the secondary damage will be rapid and widespread, leading to the removal of the coating off a large portion of the nominal contact surface. That process can influence the spread of results in an erratic manner.

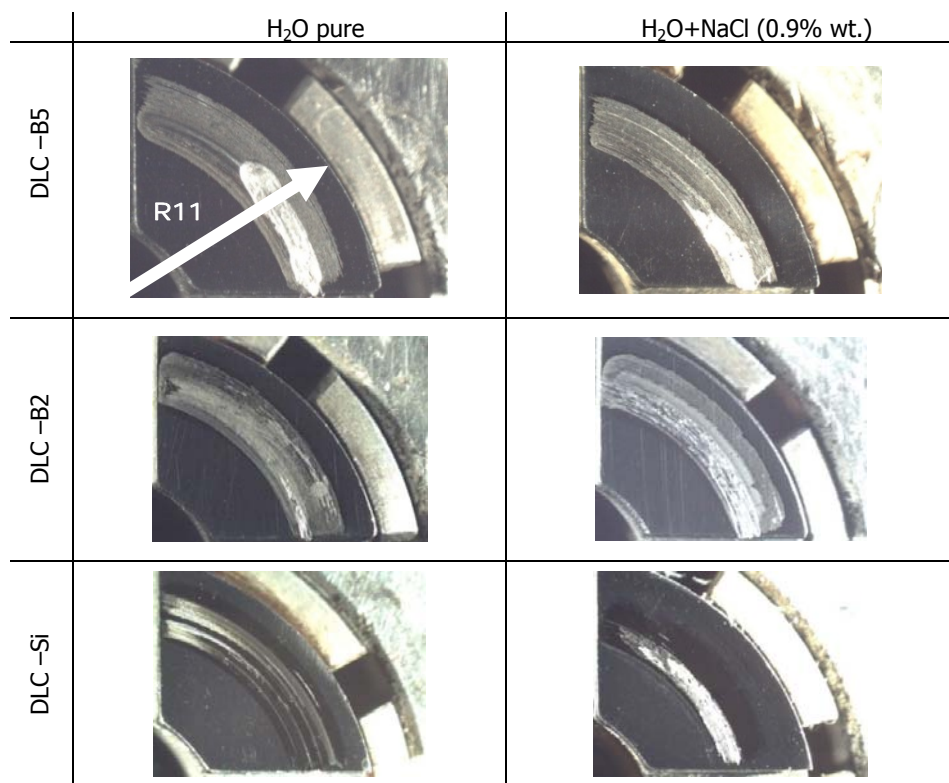


Fig. 3. Images of critically damaged lower specimens' face segments after completed wear tests. Direction of sliding – clockwise

Rys. 3. Widok krytycznie uszkodzonych powierzchni dolnych próbek – po zakończonym teście. Zwrot ruchu ślizgowego – zgodny z ruchem wskazówek zegara

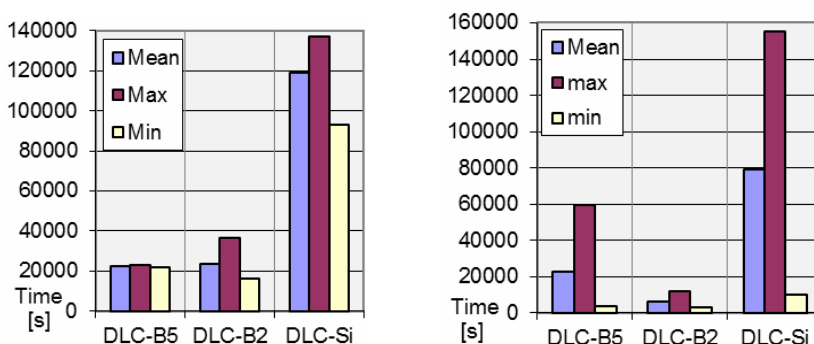


Fig. 4. Results of wear endurance tests on a PT-3 tribometer for coatings lubricated with pure water (left) and medical saline solution (right)

Rys. 4. Wyniki testów zużyciowych przeprowadzonych na tribometrze PT-3 dla powłok smarowanych czystą wodą (po lewej) oraz solą fizjologiczną (po prawej)

EXPERIMENTAL PROCEDURE – RECIPROCATING SLIDING

A concentrated contact (ball-on-flat block) reciprocating sliding system was used for the investigation of the wear of coatings. In this research, the test rig TPZ-1 [L. 13] was used. A schematic illustrating of the principle operation is shown in **Fig. 5**. The ball is fixed in a vertical plunger guided in a set of low friction linear ball bearings. The load is gravitational and constant throughout the test. The lower specimen in the form of a rectangular bar is attached to a sleigh carrying the specimen holder, lubricant container, and thermal control equipment. The whole assembly is also supported on linear ball bearings and driven by a crank mechanism.

Wear in concentrated contacts is intensive and ‘centred’ due to high contact stress. Published research shows that the type of contact can be responsible for the ‘flattening’ of differences between different materials, especially coated materials [L. 27]. At the same time, in the ball-on-disc configuration, the wear mark is well defined and concentrated in a small area, which helps the examination the process. In the tribometer used, the ball can be removed for examination and reinserted in the same position to continue the test.

The coated ball specimens – balls ($\varnothing 10$ mm, AISI 440-C steel) and blocks (X30Cr13 steel) – along with other components of the test system were thoroughly cleaned and degreased. The process was repeated each time the specimens were removed from the rig for microscopic examination and then reseated in the holders for continued testing. Distilled water and medical saline solution (0.9% NaCl in water) were used as lubricants.

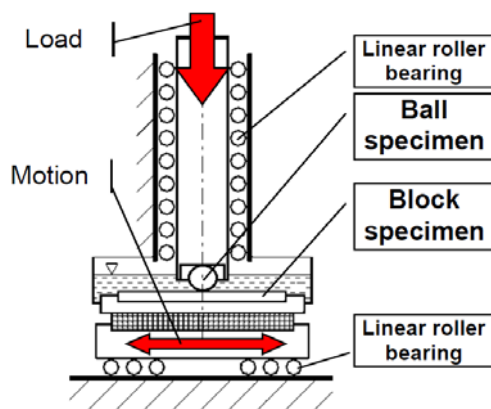


Fig. 5. Principle of operation of the TPZ-1 tribometer

Rys. 5. Zasada działania tribometru TPZ-1

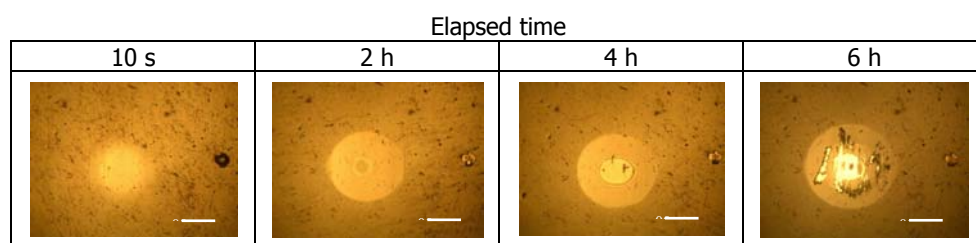


Fig. 6. Wear scar development on a DLC + B 2% coated steel ball lubricated with medical saline solution as seen in optical microscopy.

Rys. 6. Rozwój śladu zużycia na kuli stalowej pokrytej DLC z dodatkiem 2% boru, smarowanej solą fizjologiczną – obrazy z mikroskopu optycznego

The sliding velocity was constant through most of the stroke, except for the vicinity of reversing points and set at 0.08 ± 0.02 m/s. A 40 mm sliding stroke was used and the initial Hertzian contact stress was equal to 1 GPa. Tests were run at ambient temperature. The course of wear of the ceramic coating deposited on the specimen was evaluated quantitatively and qualitatively by microscopic observation. The amount of wear was measured as surface area of the wear mark on the ball (**Fig. 6**). A minimum of 3 repetitions was done for each coating variant.

RESULTS AND DISCUSSION

The course of wear was typically a very rapid increase of the wear scar on the ball surface in the initial test phase, when contact stress was highest. In the latter course of the test, the wear rate would decelerate due to the transition from concentrated (point) to distributed contact; however, with a prolonged test time,

the combined action of coating's thickness decrease and fatigue at the coating's bonding after a certain time, the coating would finally be perforated. The counter specimen wear was negligible, due to distribution over a large area as compared to the ball. Wear marks on the balls were elliptical in shape with the longer axis perpendicular to the direction of sliding. In rare cases, circular wear marks were observed. Wear was measured by digital photography analysis of the surface area of the wear mark.

Several wear forms were observed: scratches, cracks, delaminating, pits, and steel substratum exposure. Some of wear observed can be contributed to imperfections in coatings, while the deepest grooving could be caused by debris in the contact zone. The presence of defects, which reduce the load-carrying portion of the surface of contact, usually accelerates mild wear. The rapid growth occurs only when the destruction of the coating is extensive enough to expose the substrate.

Table 3. Average surface area on modified DLC coated steel balls (μm^2) at predefined time intervals in TPZ-1 reciprocating linear sliding tribometer

Tabela 3. Średnie wartości pola powierzchni śladu wytarcia na kulkach stalowych powlekanych modyfikowanym DLC (μm^2) badanych na tribometrze TPZ-1. Pomiary w ustalonych odstępach czasowych

Coating type	Lubricant type	Test time [s]			
		60	600	7 200	36 000
DLC-B5	H ₂ O pure	39 593 (10 156)	46 408 (11 127)	65 249 (7 698)	<u>99 067*</u> (10 299)
	H ₂ O+NaCl (0.9% wt.)	32 304 (3 502)	40 744 (6 556)	74 008 (8 589)	Fail**
DLC-B2	H ₂ O pure	47 254 (14 051)	54 061 (17 658)	66 836 (17 303)	96 455 (8 046)
	H ₂ O+NaCl (0.9% wt.)	38 696 (4 735)	54 429 (17 376)	<u>69 693*</u> (7 819)	Fail**
DLC-Si	H ₂ O pure	43 547 (6 442)	52 494 (5 140)	65 521 (4 294)	79 202 (4 360)
	H ₂ O+NaCl (0.9% wt.)	41 126 (5 662)	60 302 (23 571)	<u>99 488*</u> (11 320)	Fail**

* Some of the repetitions made resulted in coating's failure

** All of the repetitions made resulted in coating's failure

– Standard deviation for each time step given in brackets

In all three coatings tested, the use of medical saline as a lubricant accelerated wear and decreased the coatings' endurance (**Table 3**). The lubricant also caused slower wear mark development at an early stage of the test as compared to pure water lubrication, and the trend was reversed in later stages of the test run. The effect was noticed on all three coatings tested. In saline lubricated sliding, the wear of boron doped DLC is slower than in the silicon-doped variety. The results of the tests are inconclusive as to the influence of the



concentration of boron on wear resistance under these conditions. None of the coatings survived the 36 000 s test when lubricated with saline solution. In case of the tests run with pure water lubrication, the wear profiles of all three types of coatings are similar. The rate of wear mark growth on boron doped DLC is slightly slower in the case of a 5% concentration of boron in the coating at an early stage of the test, but the difference is nullified towards the end of the test.

The tests were conducted in 'self-mated' pairs (same coating on both sides of the friction interface). It is unlikely that material transfer played a significant role as a wear-controlling factor, which can be a case in sliding systems comprised of one DLC coated body in contact with an uncoated surface.

The results of both types of tests indicate that there is a definite influence of both factors taken into consideration on the kinetics of the wear of the model contact. There is a noticeable influence of additives on the mechanical properties of DLC coatings manifested as resistance to wear. There is a strong influence of salt content in lubricating water on the result of the experiment. It is clear that, in tests with distilled water lubrication, all coatings would wear more slowly than in the case of saline solution lubrication. The incidental results of the greater wear resistance of coatings lubricated with saline solution as compared to pure water lubrication in tests on the PT-3 tribometer should be regarded as a typical spread to tribological testing. The statement can be substantiated by the results obtained in concentrated contacts in which the presence of salt in the lubricant would radically accelerate wear on all specimens. Similar conclusions could be drawn from tests on steel coated with thin film ceramics other than DLC [L. 5].

The detailed reason for the difference in wear rate related to the presence of NaCl remains still to be investigated; however, at least two mechanisms can be named as potentially responsible for the phenomenon. The first is the electrochemical influence of electrolyte in the presence of a metal-carbon junction on the steel substrate with intermediate priming layers. The coating is both thin and porous, so the conductive fluid can penetrate the coating's imperfections. Moreover, in the contact zone, there is a high contact stress present. All these factors can lead to electrochemical deterioration of the coatings' bonding to the substrate, decrease adhesion, and an acceleration in the delamination of the coating. Secondly, a mechanical action of a third body at a micro level is possible due to flash temperatures at contacts with asperities causing local evaporation of water and the formation of microscopic salt crystals, which could locally act as abrasive particles. That process can play a more important role in tests with distributed contact in which the evacuation of debris from the friction zone is more difficult than in concentrated contacts.

Comparison of adhesion, hardness, and the thickness of coatings with wear tests does not indicate simple correlation. In the presented case, the mutual influence of these factors and the type of lubricant did not emerge in an obvious



fashion. In distributed contact tests (PT-3 tribometer), the hardness coating performs best (Si doped), despite the lowest adhesion to the substrate. Again, that result does not correlate directly with concentrated contact test results that show lower wear in boron doped coatings when lubricated with saline solution; however, the endurance result is identical for all coatings tested. The silicon doped coating seems to perform better with pure water lubrication, delivering a wear mark size comparable to boron doped coatings (both performing similarly), but the overall endurance result is identical for all three coating types. There is no significant difference between the wear resistance of boron-doped coatings at the element concentration levels of 2% and 5% for both lubricants used.

In a typical ball-on-flat test (TPZ-1 rig), there was a steady growth of the wear mark on the ball's surface, which would later develop from a flat, uniform area into a system of cracks and dents of gradually increasing size until a large perforation of the coating is reached and the test is discontinued. The progress of wear in a distributed contact test (PT-3 rig) was comparable to the concentrated contact test. The initial mild wear would lead to coating's discolouration and slight scratching, but the adhesion was always sufficient to prevent the removal of larger fragments of a coating under the action of tangential forces at that stage. In the further progress of the tests, the minute wear marks would accumulate and join, leading to a gradual removal of the layers of coating and finally to perforation followed by metallic contact in the friction zone.

A general conclusion emerging from the data gathered is that, in order to evaluate the characteristics of DLC type coatings, it is necessary to run tests in several configurations in order to obtain credible results that are useable as a basis for technical applications.

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Streszczenie

W artykule przedstawiono wyniki kompleksowych badań zużycia powłok DLC domieszkowanych borem (B) oraz krzemem (Si). Zadanie zrealizowano w dwóch konfiguracjach styku ślizgowego: płaszczyzna – płaszczyzna (styk o kształcie pierścieniowym, ruch jednokierunkowy) i kula – płaszczyzna (ruch prostoliniowy, zwrotny). Jako smary wykorzystano wodę destylowaną i sól fizjologiczną. Analiza porównawcza wyników wykazała różnice w przebiegu inicjacji zużycia oraz w trwałości powłok w zależności od składu chemicznego powłoki, substancji smarowej i geometrii styku. Wyniki wskazują na wpływ wszystkich uwzględnionych czynników na zużycie. We wszystkich przypadkach zastosowanie soli fizjologicznej jako smaru skutkowało przyspieszonym zużyciem.



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