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DETERMINING THE TRIBOLOGICAL PROPERTIES OF DIAMOND-LIKE CARBON COATINGS LUBRICATED WITH BIODEGRADABLE CUTTING FLUIDS

The aim of the study was to determine the effectiveness of the biodegradable cutting fluid used instead of classical, usually toxic. This paper presents the results of tribological studies of a-C:H coatings formed on HS6-5-2C steel by plasma-assisted chemical vapour deposition. The coating structures were examined using a JSM-7100F SEM. The coating microhardness was measured with a Matsuzawa tester. The surface texture analysis was performed before and after the tribological tests with a Talysurf CCI Lite optical profiler. The tribological properties were investigated using a T-01 M tester and a T-17 tester. The tests were carried out under dry friction conditions and lubricated friction conditions using a lubricant with zinc aspartate. The test results show that the layer formed at the interface prevented the moving surfaces. The investigations discussed in this paper have contributed to the development of non-toxic and environmentally-friendly manufacturing because of the use of biodegradable cutting fluid and thin, hard coatings.

Keywords: biodegradable cutting fluid, diamond-like carbon, friction, wear

1. Introduction

Diamond-like coatings were first discovered by Aisenberg and Chabot. Amorphous carbon consists of a mixture of sp^3, sp^2, and even sp^1 bonded atoms, with hydrogen either present or absent. Diamond-like coatings can have a variety of structures. Their properties are dependent on the deposition methods and the parameters used, with another important factor being the graphite sp^2 to diamond sp^3 ratio. The coatings can be doped with metals (W, Ti, Nb, Cr, V, Co, Mo) and non-metals (H, Si, F, N, O, P, B), which improves their properties while maintaining their amorphous character [1].

Recent years have witnessed rapid advances in the research on thin diamond-like carbon coatings applied by chemical vapour deposition (CVD) or physical vapour deposition (PVD). Because of their excellent tribological properties, including low friction and high wear resistance, as well as high stability, corrosion resistance and hardness, DLC coatings are increasingly common in many industries. Like other state-of-the-art materials, they need to have good mechanical, physical, chemical and fabrication properties to ensure long service life and reliability of products [2-8]. In addition to the exceptional mechanical and tribological properties, the materials are characterised by low and negative electron affinity, excellent thermal conductivity and a very low coefficient of thermal expansion. Diamond-like carbon coatings have properties similar to those of diamond [9-10].

It is important to note that diamond-like carbon coatings can be deposited at very low temperatures [10]. There is practically no limitation on the type of material used for the substrate [11].

Reference [3] analyses the effect of the tungsten content on the properties of DLC coatings deposited on steel to be applied in the chemical industry. It compares a-C:H coatings deposited by PACVD with a-C:H:W coatings deposited by PVD. It also studies the relationships between the type, elemental composition and surface texture of the coatings and their corrosion and tribological properties. Tests conducted under dry friction conditions and under lubricated friction conditions with 1-butyl-3-methylimidazolium tetrafluoroborate as a lubricant show that machine parts with DLC coatings used in friction pairs exhibit better tribological properties than elements without such coatings. It is reported that the application of the ionic liquid improves the friction properties of systems, and the presence of tungsten in DLC coatings contributes to an improvement of tribological properties and a slight decrease in corrosion resistance.

The research discussed in Ref. [4] involved tribological studies of a-C:H coatings under dry friction conditions and under lubricated friction conditions using base oil or gear oil. The synthetic lubricants used in the tests were responsible for a reduction in the coefficient of friction. Reference [6] deals with a comprehensive analysis of the tribological properties of systems with DLC coatings under dry friction conditions and

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when lubricated with a polyalphaolefin or an ionic liquid. The results confirm the effectiveness of both DLC coatings and ionic liquids applied to improve tribological properties.

DLC coatings characterised by excellent properties such as good corrosion resistance and high hardness can be used for many industrial applications, e.g. on cutting tools used in machining, which operate both in dry and lubricated contact with a cooling/cutting fluid [11]. Cutting tools with a long service life must be made of a material that is harder than the workpiece material [12]. Machining processes also require suitable cooling/cutting fluids to reduce friction, cool the workpiece and remove chips from the cutting area. Cutting fluids are also responsible for reducing tool wear, improving surface quality of the workpiece, protecting it from corrosion, minimizing shear forces and thus saving energy. In conventional machining, there are three basic forms of energy: mechanical, electrical and thermal [12-15].

Modern cutting fluids need to be safe and biodegradable. Unfortunately, most cutting fluids pose a threat to human health and the environment. Occupational exposure to cutting fluids can have a number of adverse health effects including allergic reactions, infections, skin and eye irritation and even cancer. Cutting fluid waste may be responsible for the contamination of surface and ground water. Thus, it is vital that used cutting fluids be properly disposed of, recycled and/or reused [14-16].

Our current research looks at environmentally-friendly coolants and lubricants as well as coatings with superior anti-wear properties. It is in line with the regulations concerning environmental protection and environmentally-friendly solutions for industry.

This study focuses on the performance of biodegradable zinc aspartate, which is to replace toxic zinc dialkyldithiophosphates (ZDDPs). Because of their good anti-wear and anti-corrosion properties, ZDDPs are commonly used as additives in lubricants. Zinc aspartate, which is also a zinc-based compound, has so far been used mainly in medical and pharmaceutical applications [17].

2. Materials and Methods

The aim of the wear tests was to study the effects of a biodegradable cutting fluid on the tribological performance (coefficient of friction) of the analysed friction configurations:

- T-01M ball-on-disc tester – balls made of 100Cr6 steel was pressed against a test sample HS6-5-2C tool steel with and without an a-C:H-type diamond-like carbon coating,
- T-17 pin-on-plate tribometer – pins made of HS6-5-2C were in sliding contact with a test sample made of HS6-5-2C tool steel with and without an a-C:H coating.

2.1. Substrate and Coating Materials

The tribological tests were conducted using a biodegradable cutting/cooling fluid based on demineralised water (DEMI) containing 5% vol. aqueous solution of zinc aspartate. This metal cutting fluid is composed of alkanolamine borate, biodegradable polymer containing zinc aspartate and water. Table 1 presents the parameters of the demineralised water.

TABLE 1

Parameters of the demineralised water at 25°C

pH	Conductivity, $mS \cdot cm^{-1}$	maximum resistivity, $M\Omega \cdot cm$
approx. 5.2-6.0(5.0-7.2)	$5.5^{-10} \cdot 10^{-5}$ (1.42 μS/cm)	18.2

The samples made of HS6-5-2C tool steel, with and without a-C:H DLC coatings, were tested in configurations with 100Cr6 steel balls or HS6-5-2C steel pins. Diamond-like carbon coatings have properties similar to those of diamond. Mechanical properties of diamond and a-C:H-type diamond-like carbon coatings shown in Table 2 [10]. The a-C:H-type diamond-like carbon DLC coatings were produced by plasma-assisted chemical vapour deposition (PACVD) at <250° on high-speed tool steel HS6-5-2C. The composition of HS6-5-2C steel is shown in Table 3.

TABLE 2

Mechanical properties of diamond and a-C:H-type diamond-like carbon coatings [9]

Material	Form	Density, g/cm^3	Covalent bonds	Young's modulus, GPa	Hardness, GPa
a-C:H	Films	~2.2	Intermediate sp^3	100-300	10-30
Diamond	Bulk, films	~3.5	100% sp^3	1000	100

HS6-5-2C steel is designed to operate at high temperatures; it can be subjected to heat treatment: tempering at 1190-1230°C and quenching at 550-650°C. Its hardness after heat treatment at 500-550°C is 65 HRC.

2.2. Methods

2.2.1. Structural and chemical analysis (before the tribological tests)

A JSM 7100F scanning electron microscope was employed to examine the surface topography and the cross-sections of the

TABLE 3

Composition of HS6-5-2C steel

Element	C	Mn	Si	P	S	Cr	Ni	Mo	W	V	Co	Cu
Percentage, %	0.82-0.92	≥0.4	≥0.5	≥0.03	≥0.03	3.5-4.5	≥0.4	4.5-5.5	6-7	1.7-2.1	≥0.5	≥0.3

DLC coatings. An EDS analysis was performed to identify the elements present in the a-C:H coating. The mechanical properties of the DLC coatings were determined on the basis of the microhardness measurements conducted with a Matsuzawa tester. The effect of the substrate on the measurement of the coating hardness was minimised by assuming that the indentation depth could not exceed one tenth of the coating thickness. A Vickers indenter was used and it was subjected to a load of 98.07 mN. The surface texture of the discs, with and without DLC coatings, was analysed before and after the tribological tests using a Talysurf CCI Lite optical profiler.

2.2.2. Tribological tests

The tribological tests were conducted using a T-01M ball-on-disc system (Fig. 1a) and a T-17 pin-on-plate system (Fig. 1b). In the T-01M tester, a rotating ball made of 100Cr6 steel (diameter = 10 mm) was pressed against a test sample (HS6-5-2C tool steel with and without an a-C:H-type diamond-like carbon coating), applying a constant sliding velocity of 0.1 m/s and a load of 10 N. The sliding distance was 1000 m. In the T-17 tribometer, pins made of HS6-5-2C steel (diameter = 9 mm) were in sliding contact with a test sample made of HS6-5-2C tool steel with and without an a-C:H coating. The tests were conducted at a frequency of 1 Hz, an amplitude of 12.7 mm and a load of 100 N, all being constant. The number of cycles was 10 000. The specimens were tested under dry friction conditions and under lubricated friction conditions using a biodegradable cooling/cutting fluid based on DEMI demineralised water containing 5% vol. aqueous solution of zinc aspartate. The tests were performed in laboratory conditions at a relative humidity of 50 ±5% and a temperature of 23 ±1°C.

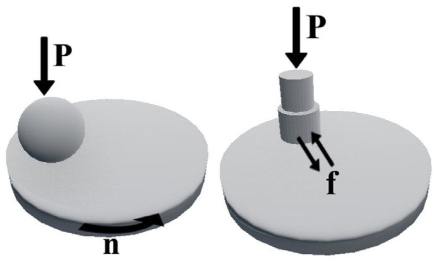

Fig. 1. Schematic diagrams of a) the ball-on-disc configuration and b) the pin-on-plate configuration

3. Results and discussion

3. 1. Structural and chemical analysis (before the tribological tests)

Figure 2 shows SEM images of the diamond-like carbon coating. Fig. 2a) illustrates a cross-section of a 3.68 µm thick a-C:H coating deposited on the HS6-5-2C steel substrate. As revealed by the EDS analysis (Fig. 2b), there was a chromium interlayer between the substrate and the DLC coating, which contributed to good coating adhesion [3].

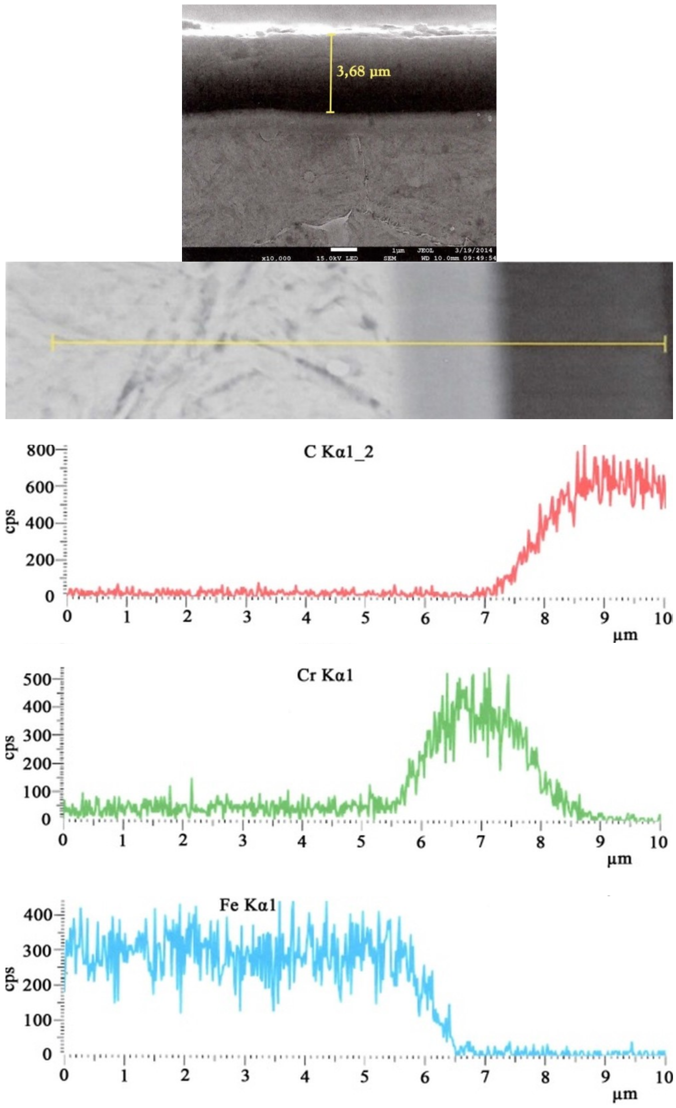

Fig. 2. a) SEM cross section and b) EDS analysis of the a-C:H coating

The results of the Vickers hardness tests presented in Table 4 indicate that the a-C:H coating is nearly 3-times higher than the steel substrate.

TABLE 4

Hardness of the substrate and the a-C:H-type DLC coating

Material	Hardness, $HV_{0.01}$	Standard Deviation
Substrate	771	18.5
a-C:H	1906	29

The figures below show the surface topography and roughness profiles for an uncoated HS6-5-2C steel disc (Fig. 3) and an HS6-5-2C steel disc with an a-C:H coating deposited by PACVD (Fig. 4). The results obtained for the coated and uncoated specimens were the reference data after the friction tests.

There was a clear difference in the surface texture profiles between the uncoated HS6-5-2C steel discs and the HS6-5-2C steel discs coated with a-C:H (Fig. 4); however, the irregularities were smaller for the a-C:H coated specimens.

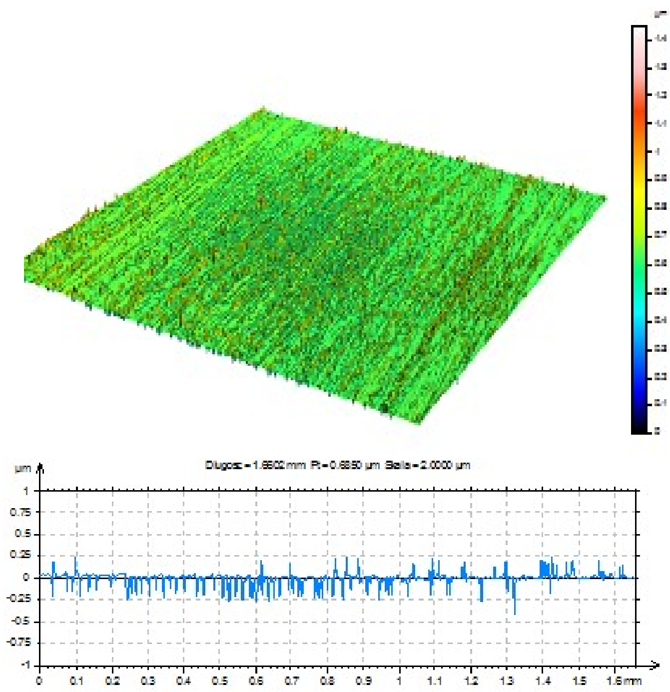

Fig. 3. HS6-5-2C steel disc: a) surface texture, b) roughness profile

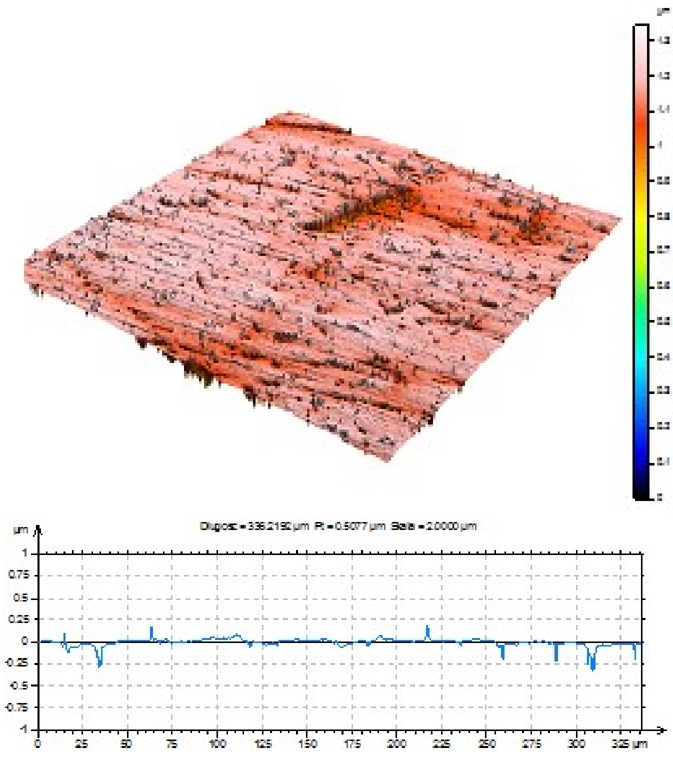

Fig. 4. HS6-5-2C steel disc with an a-C:H coating: a) surface texture, b) roughness profile

3.2. Tribological tests

Figure 5 shows the coefficients of friction obtained for the HS6-5-2C steel/100Cr6 steel and a-C:H coating/100Cr6 steel systems tested under dry friction conditions (TDF) and lubricated friction conditions using a cooling/cutting fluid (CF). The results reported for dry friction conditions were the reference data.

The lowest coefficient of friction was recorded for the a-C:H coating/100Cr6 steel configuration lubricated with the cooling/cutting fluid and it was $\mu \cong 0.027$. The highest value was obtained for the HS6-5-2C steel/100Cr6 steel pair operating under dry friction conditions ($\mu \cong 0.56$).

Analysing the diagram above, we can conclude that after the tribological tests the coefficient reported for the a-C:H coating was lower than that for HS6-5-2C steel, both in dry and lubricated contact.

The diamond-like coating (a-C:H) and the biodegradable cutting fluid used in the study had a considerable effect on friction; they caused a reduction in the friction coefficient.

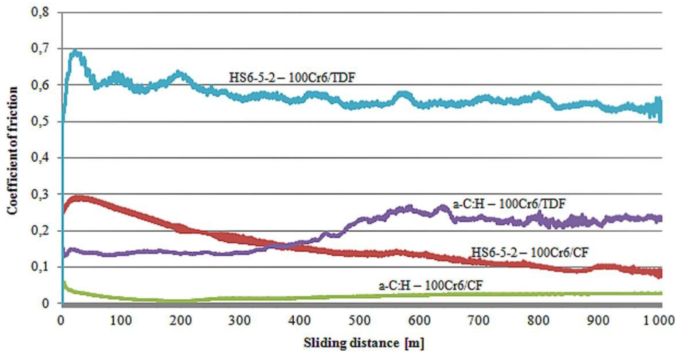

Fig. 5. Coefficient of friction for the HS6-5-2C steel-100Cr6 steel and a-C:H coating–100Cr6 steel systems. Sliding distance: s = 1000 m. Load: P = 10N. T-01M ball-on-disc tester

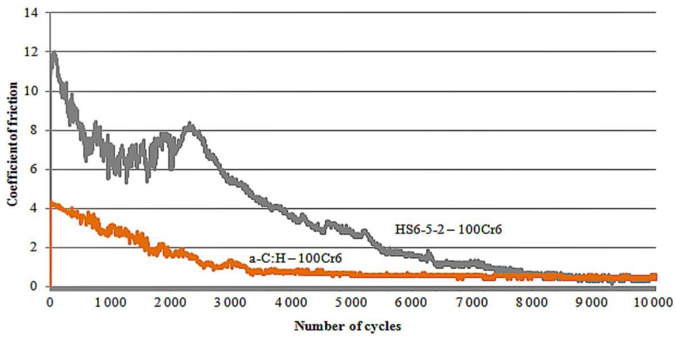

Fig. 6. Coefficient of friction for the HS6-5-2C steel–HS6-5-2C steel and a-C:H coating–HS6-5-2C steel systems. Number of cycles: c = 10 000. Load: P = 100N. T-17 pin-on-plate tester

Figure 6 shows the relationship between the coefficient of friction and the number of cycles after the tribological tests performed with the T-17 tester. The coefficient of friction reported for the a-C:H coating/HS6-5-2C steel configuration in lubricated contact was lower ($\mu \cong 0.4$). For the HS6-5-2C steel/HS6-5-2C steel pair, the coefficient was higher ($\mu \cong 0.7$).

This is due to greater wear of the ball because the a-C:H coating is very hard. Table 5 shows the wear mesurement and results after the tribological tests.

The tribological tests were conducted under dry and lubricated conditions. Two parameters were considered: volume loss and wear rate. The lowest values (50% lower) were reported for discs coated with a-C:H.

TABLE 5

Wear mesurement and results after tribological tests, ball-on-disc tester

Parameters	HS6-5-2C–100Cr6/TDF	a-C:H–100Cr6/TDF	HS6-5-2C–100Cr6/CF	a-C:H–100Cr6/CF
Wear track diameter d, mm	14	14	10	10
Sliding distance S, m	1000	1000	1000	1000
Integrated area across the wear track A, mm^2	0,1425	0,0114	0,0863	0,0063
Wear volume loss V, mm^3	6,2657	0,5022	2,7111	0,1971
Wear rate W, mm^2/m	0,0063	0,0005	0,0027	0,0002

3.3. Structure after the tribological tests

Figures 7-12 illustrate the surface topography and roughness profiles obtained for the discs tested under dry friction conditions and under lubricated friction conditions with the cooling/cutting fluid.

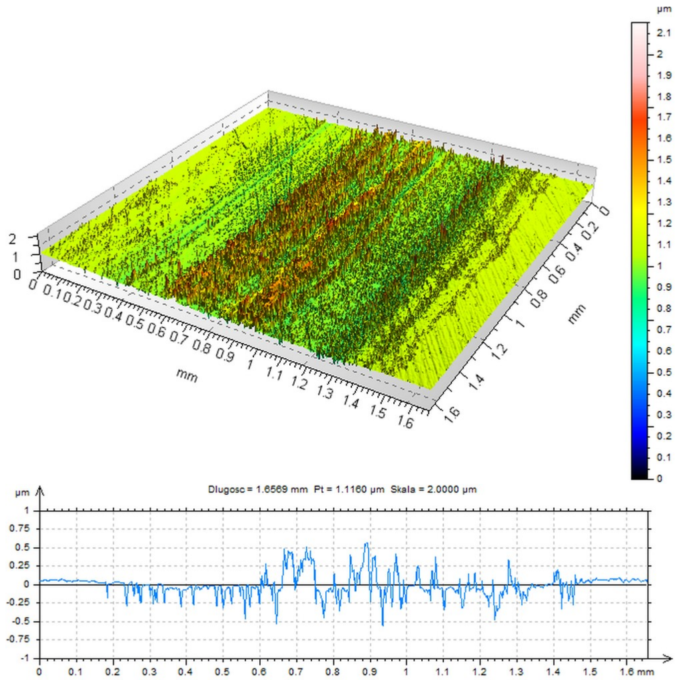

Fig. 8. Surface texture of an HS6-5-2C steel disc in lubricated contact with a 100Cr6 steel ball: a) surface topography, b) roughness profile. T-01M tester

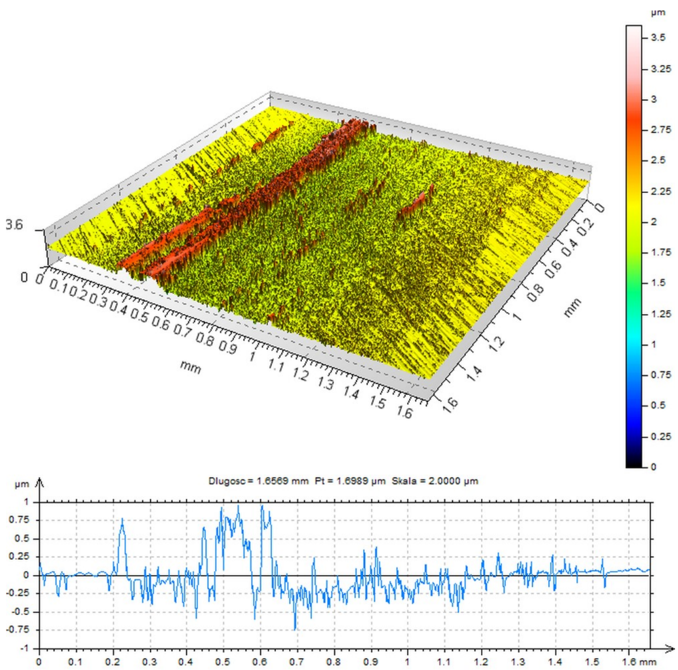

Fig. 7. Surface texture of an HS6-5-2C steel disc in dry contact with a 100Cr6 steel ball: a) surface topography, b) roughness profile. T-01M tester

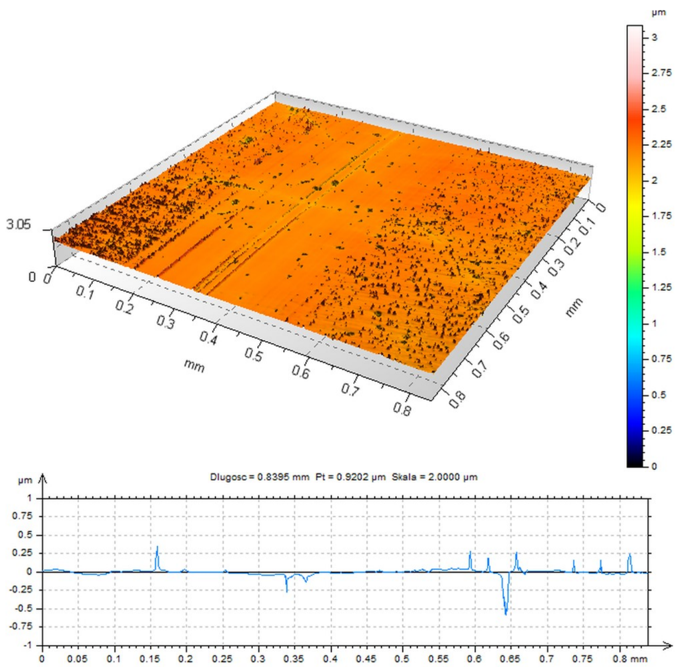

Fig. 9. Surface texture of an a-C:H coating in dry contact with a 100Cr6 steel ball: a) surface topography, b) roughness profile. T-01M tester

From the comparative analysis of the roughness profiles obtained for the different discs it is clear that in the case of the HS6-5-2C steel disc/steel ball configuration the peaks were lower under lubricated friction conditions rather than under dry friction conditions. For the a-C:H-coated disc, the grooves were less deep when the system was lubricated with the cooling/cutting fluid rather than in dry contact. Comparing the surface roughness profile of the a-C:H-coated steel disc with that of the uncoated disc, both lubricated with the cutting fluid, we can see that the a-C:H-coated surface had fewer peaks and valleys because the profile is smoother. This indicates that the a-C:H coating was harder and more resistant to wear.

Table 6 shows the most important roughness parameters of the HS6-5-2C steel discs before and after the tribological tests.

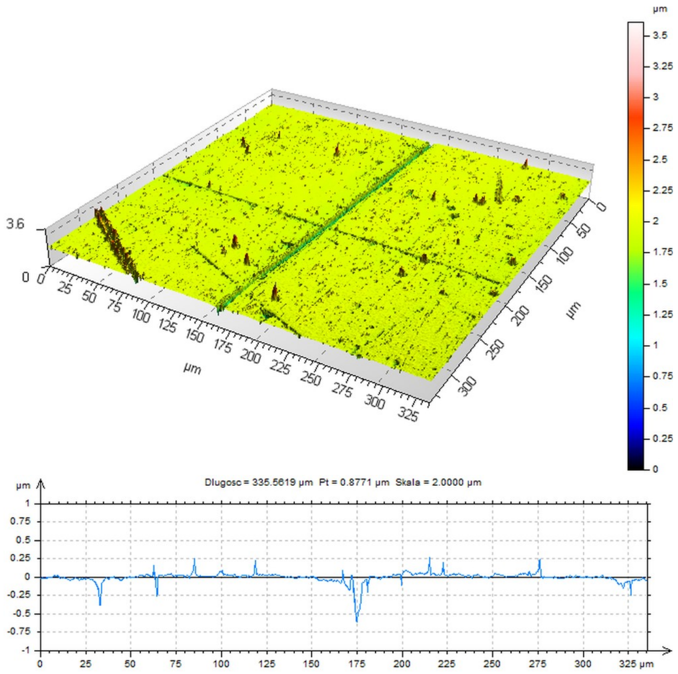

Fig. 10. Surface texture of an a-C:H coating in lubricated contact with a 100Cr6 steel ball: a) surface topography, b) roughness profile. T-01M tester

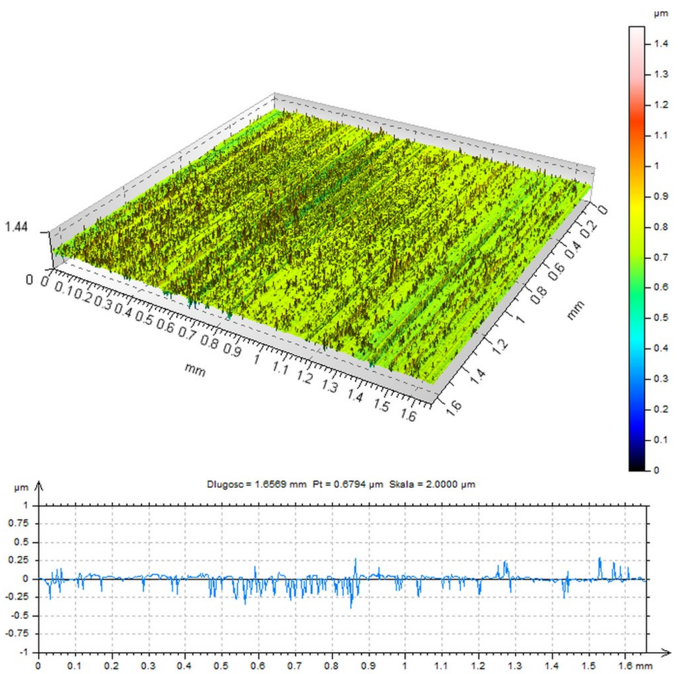

Fig. 11. Surface texture of an HS6-5-2C steel disc in lubricated contact with an HS6-5-2C steel pin: a) surface topography, b) roughness profile. T-17 tester

Comparing the surface texture parameters obtained for the discs made of HS6-5-2C steel under dry conditions with the surface texture parameters reported for friction under wet conditions, we can see that the values of the parameters:

- Sa, Sq, Sp, Sv, Sz Ssk – increased,
- Sku – decreased only after the tribological test performed with the T-17 tester.

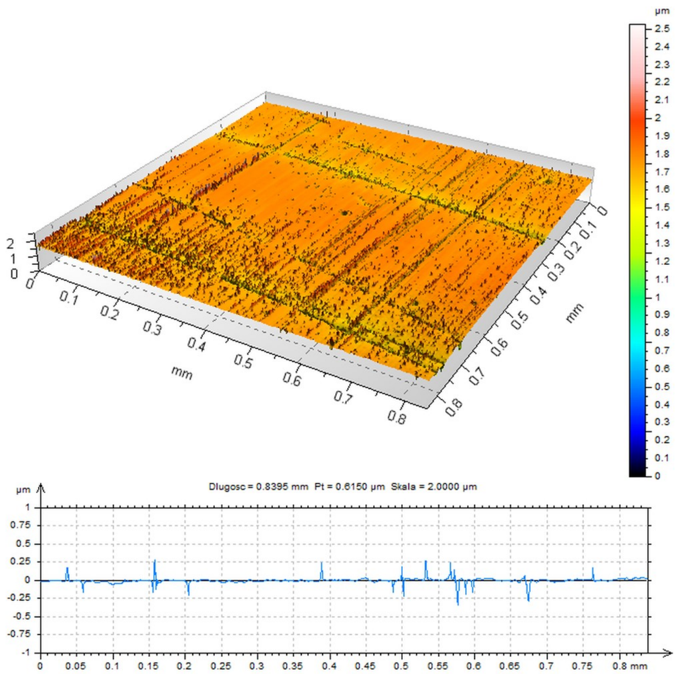

Fig. 12. Surface texture of an a-C:H coating in lubricated contact with an HS6-5-2C steel pin: a) surface topography, b) roughness profile. T-17 tester

The analysis of the roughness parameters showed that when the cutting fluid was present, some smoothing of the disc surface was observed.

Table 7 shows roughness parameters of the a-C:H-coated disc before and after tribological testing.

In the case of the HS6-5-2C discs coated with a-C:H, however, the values of the surface texture parameters before and after the tribological tests under wet conditions:

- Ssk – increased after the tribological test performed with the T-17 tester,
- Sa, Sq, Sp, Sv, Sz, Ssk, Sku - decreased,
- Sa – remained unchanged only after the tribological test performed with the T-01 tester [18-22].

Evaluating the results obtained after the tribological tests with the use of cutting fluid was observed smoother surface of the disc.

Corrosion resistance was tested in accordance with the PN-92-M-55798 standard using the Ford method. A biodegradable cutting fluid was compared with a coolant containing mineral oil. The tests involved placing cast iron chips on filter paper and soaking them in each fluid for 2 hours. The state of the filter papers was examined after the chips were removed. The degrees of rust observed for the biodegradable cutting fluid and the coolant containing mineral oil were 1 (negligible corrosion) and 2 (light corrosion), respectively.

TABLE 6

Surface roughness parameters of the HS6-5-2C steel discs, HS6-5-2C steel pins and 100Cr6 steel balls

Test	Sample		Surface roughness parameters						
			Sa, µm	Sq, µm	Sp, µm	Sv, µm	Sz, µm	Ssk, -	Sku, -
Before test	HS6-5-2C	disc	0.04	0.07	0.84	0.62	1.45	–1.03	8.94
	100Cr6	ball	0.10	0.13	0.32	1.41	1.74	–1.11	4.75
After test	TDF HS6-5-2C – 100Cr6 T-01M	disc	0.15	0.24	1.53	2.07	3.61	1.70	7.43
		ball	0.07	0.11	0.78	1.07	1.85	–2.49	13.89
	CF HS6-5-2C – 100Cr6 T-01M	disc	0.09	0.13	1.09	1.06	2.15	0.69	6.57
		ball	0.07	0.08	0.72	0.55	1.27	–0.43	3.73
	CF HS6-5-2C – HS6-5-2C T-17	plate	0.04	0.07	0.74	0.71	1.46	–0.91	10.49
		pin	0.03	0.07	0.60	1.02	1.62	–0.30	10.12

TABLE 7

Surface roughness parameters of the a-C:H-coated discs, HS6-5-2C steel pins and 100Cr6 steel balls

Test	Sample		Surface roughness parameters						
			Sa, µm	Sq, µm	Sp, µm	Sv, µm	Sz, µm	Ssk, -	Sku, -
Before test	a-C:H coating	disc	0.04	0.06	0.19	1.16	1.34	–3.26	21.7
	100Cr6	ball	0.10	0.13	0.32	1.41	1.74	–1.11	4.75
After test	TDF a-C:H – 100Cr6 T-01M	disc	0.03	0.06	0.88	2.21	3.09	–4.30	104.07
		ball	0.23	0.29	1.39	1.63	3.02	–0.16	3.10
	CF a-C:H – 100Cr6 T-01M	disc	0.04	0.08	1.69	1.92	3.61	–1.78	39.08
		ball	0.24	0.30	1.74	1.03	2.77	0.39	3.39
	CF a-C:H – HS6-5-2C T-17	plate	0.05	0.09	0.79	1.74	2.53	–3.17	32.52
		pin	0.09	0.13	1.01	0.88	1.88	–0.19	5.21

4. Conclusion

The investigations concerning the application of diamond-like carbon coatings and cutting fluids are still in progress. Their goal is to provide a modern approach to materials used in tribological systems.

The results of the chemical composition analysis indicate that the a-C:H coating contains carbon and chromium constitutes the interlayer at the interface between the substrate and the DLC coating. After the deposition of a DLC coating, the hardness of a steel sample increases by more than 2.5-fold.

The analysis of the surface topography conducted with the optical profiler indicated that thea-C:H coating had better surface texture parameters than steel. The a-C:H-coated surface had fewer peaks and valleys, the profile was smoother. After the tribological tests, the grooves and the wear track on the surface of the DLC-coated disc were smaller than those on the uncoated steel disc.

Diamond-like carbon coatings deposited on steel components exposed to tribological wear provide excellent wear protection. These properties are enhanced when a biodegradable cutting fluid is added to a tribological system.

The cutting fluid containing zinc aspartate used in the tests ensured stable performance of the tribotechnical system and contributed to a reduction in the coefficient of friction. It effectively performed its predetermined function as a coolant and lubricant; it was also safe for the operator and the environment.

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