ISSN 0208-7774 TRIBOLOGIA 5/2019 p. 59–69
DOI: 10.5604/01.3001.0013.6563

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TRIBOLOGICAL PROPERTIES OF DIAMOND-LIKE CARBON COATINGS AT FRICTION JOINTS LUBRICATED WITH IONIC LIQUID

WŁAŚCIWOŚCI TRIBOLOGICZNE POWŁOK DIAMENTOPODOBNYCH W WĘZŁACH TARCIA SMAROWANYCH CIECZĄ JONOWĄ

Key words: DLC coatings, wear, ionic liquids, friction.

Abstract The paper reports the study of a-C: H and a-C:H:Si diamond-like carbon coatings obtained in plasma-assisted chemical vapour deposition processes PACVD. The influence of the coatings on tribological properties of tribo-pairs under dry friction and in lubrication with synthetic oil PAO-8 and selected ionic liquid was evaluated. To perform the analysis, 100Cr6 steel samples uncoated and coated with a-C:H and a-C:H:Si were compared. Surface topography studies were performed using an atomic force microscope. Using SEM with an EDS analyser, the surface morphology of the coatings was observed, and the elements contained in the coatings were identified. Tribotests were carried out in a ball-on-disc tribotester under dry friction and with lubricants. The characteristics of the texture of the samples before and after the tribotests were determined using a confocal microscope in an interferometric mode. The test results showed that the tribo-pairs with a-C:H in lubrication with ionic liquid had the best tribological characteristics, i.e., the lowest coefficient of sliding friction.

Słowa kluczowe: powłoki DLC, zużycie, ciecze jonowe, tarcie.

STreszczenie Artykuł dotyczy badań powłok diamentopodobnych typu a-C:H oraz domieszkowanych krzemem a-C:H:Si uzyskiwanych w procesach chemicznego osadzania z fazy gazowej wspomaganych plazmą PACVD. Poddano ocenie wpływ powłok na właściwości tribologiczne węzłów tarcia w warunkach tarcia technicznie suchego oraz smarowania olejem syntetycznym PAO-8 i cieczą jonową bis(trifluorometylosulfonylo) amidem triheksylotetradecylofosfoniowym. W celu przeprowadzenia analizy porównano próbki ze stali 100Cr6 oraz pokryte diamond-like carbon coating typu a-C:H oraz a-C:H:Si. Badania topografii powierzchni wykonano na mikroskopie sił atomowych. Obserwacje morfologii powierzchni i identyfikację pierwiastków przeprowadzono przy użyciu skaningowego mikroskopu elektronowego z analizatorem EDS. Badania tribologiczne przeprowadzono na tribotesterze pracującym w skojarzeniu kula–tarcza w warunkach tarcia technicznie suchego oraz tarcia z zastosowaniem substancji smarowych. Charakterystykę struktury geometrycznej próbek przed oraz po badaniach tribologicznych wykonano mikroskopem konfokalnym z trybem interferometrycznym. Wyniki badań wskazały, że najlepszymi charakterystykami tribologicznymi – najmniejszym współczynnikiem tarcia w ruchu ślizgowym charakteryzowały się pary trące z diamond-like carbon coating typu a-C:H w warunkach smarowania cieczą jonową.

INTRODUCTION

The outer layer of friction surfaces plays an important role in initiating the course of friction and the processes accompanying friction **[L. 1]**. A variety of technological solutions have been applied so far to reduce the adverse effects of friction in the surface layer area. Diamond-like carbon (DLC) coatings, used in a number of industrial applications for their excellent tribological properties, have proven to perfectly suit the purpose. Thin and hard films are deposited by chemical vapour deposition CVD and physical vapour deposition PVD techniques, usually in the plasma assisted process **[L. 2]**. Tribological and mechanical properties of DLC coatings can be varied by

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the appropriate choice of deposition methods and film composition. Application of thin, hard coatings to friction elements of machines and devices results directly from the benefits they offer, such as a low friction coefficient and resistance to tribological wear and corrosion. These advantages contribute greatly to the increased service life of the friction pairs **[L. 3–5]**.

Three functional regions should be present in the coatings, regardless of their structural design **[L. 2, 3]**:

- The outer surface layer, which is responsible for the interaction with the environment and tribological cooperation within the tribo-pair (This layer should be strong in shear, chemically reactive and have adequate roughness.);
- The main inclusive layer, which is responsible for the hardness, flexibility, toughness, thermal stability and thermal conductivity; and,
- The interlayer between the film and the substrate, which is responsible for appropriate adhesion to the substrate and shear resistance.

In terms of their structure, diamond-like carbon coatings are classified as follows **[L. 6]**:

- a-C:H – with the hydrogen content ranging from 20 to 40% and a *sp*^{3} fraction of 30%;
- a-C – hydrogen free, with a *sp*^{3} fraction up to 30%;
- ta-C:H – with a *sp*^{3} content of ~70% and a hydrogen content from 25 to 35%, where the fraction of *sp*^{3} bonding increases with an increasing bonded hydrogen content; and,
- ta-C – hydrogen-free tetrahedral amorphous carbon having the maximum *sp*^{3} fraction that affects primarily the elastic properties.

Wear of machine working elements depends on the method of coating application, the parameters of this process and operating conditions acting on a given machine working element, such as corrosion, nominal loads in the friction node, lubricants used, corrosive and tribocorrosive interactions, the use of the machine in accordance with the specifications **[L. 8–9]**.

MATERIALS

Diamond-like carbon coatings, an a-C:H and a Si-doped a-C:H:Si obtained by the PACVD technique were the subject of the investigation. The coatings were deposited on 100Cr6 roller-bearing steel at a temperature below 250°C. The PACVD technique is a glow discharge plasma-assisted CVD process that can produce hard surface layers or layers with the desired properties. In this technique, the carbon layer is deposited by adsorbing free hydrocarbon radicals to the surface of the substrate and forming chemical bonds between the other atoms on the surface.

Tribological tests were performed under dry friction using lubricants such as a synthetic oil PAO-8 and ionic liquid tetradecyltrihexylphosphonium bis(trifluoromethylsulfonyl) amide. This ionic liquid was chosen for its good tribological properties, in particular, for the formation of fluoride and sulphide tribofilms at high loads **[L. 10, 11]**. In addition, the liquid acts as a good solvent and can be used as a base or additive to lubricants.

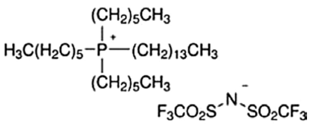
Selected properties of the lubricants used in this study are compiled in **Table 1**.

The aim of the study was to evaluate tribological properties of diamond-like carbon coatings type a-C:H and a-C:H:Si under dry friction conditions and with the use of lubricants. The thicknesses of a-C:H and a-C:H:Si coatings, approximately 1.83 μm and 1.71 μm, respectively, was determined by means of a compact Calotest CATc apparatus and optical microscope. The hardness was determined using the Anton Paar NHT3 nano-hardness tester and was 21.08 GPa for a-C:H and 14.65 GPa for a-C:H:Si.

METHODS

The topography of the analysed surfaces was examined using an atomic force microscope AFM Nanosurf ATS 204. A scanning electron microscope with an

Table 1. Structure and selected properties of lubricants used
Tabela 1. Struktura i wybrane właściwości zastosowanych smarów

Symbol	Synonym	Linear formula	Structural formula	Molecular weight [g/mol]	Density [g/cm^3]
IL	Tetradecyltrihexyl phosphonium bis(trifluoromethyl sulfonyl)amide	$[CH_3(CH_2)_5]_3P[N(SO_2CF_3)_2](CH_2)_{13}CH$		764	1.07
PAO-8	Poly-α-olefin	$C_{14}H_{28}$	$CH_2 = CH(CH_2)_{11}CH_3$	196	0,84

EDS analyser was used for the observations of surface morphology and identification of elements. The texture of the surfaces before and after tribotests was analysed using a Leica DCM8 confocal microscope.

Tribotests were performed on a TRB tribotester working in a ball-on-disk configuration. The tests were carried out in the following regime:

- Friction pairs: an uncoated 100Cr6 steel ball, 6 mm in diameter; 100Cr6 steel disks, 42 mm in diameter, uncoated, a-C:H coated and a-C:H:Si coated;
- Load: P = 10 N;
- Sliding speed: v = 0.1 m/s;
- Sliding distance: s = 1000 m;
- Relative humidity: 45 ±1%;
- Ambient temperature: T_0 = 25±1°C;
- Lubricants: a) none, b) synthetic oil: PAO-8, c) ionic liquid: trihexyltetradecylphosphonium bis(trifluoromethylsulfonyl) amide.

RESULTS AND DISCUSSION

Figures 1 and **2** show the topography of the surface layers of the a-C:H and a-C:H:Si coatings from the atomic force microscope.

The topography of the a-C: H surface deposited on 100Cr6 steel shown in **Fig. 1** confirms its good quality and the homogeneity of the coating. In **Fig. 2**, however, dispersed particles can be seen on the surface of the a-C:H:Si coating. An electron microscope was employed to map the distribution of the chemical element concentration, with the EDS spectrum and mass content of the elements for a-C: H **(Fig. 3)** and a-C:H-Si (**Fig. 4**).

Figures 3 and **4** show images of surface morphology and analysis of the chemical composition of elements in the micro-area of a 100Cr6 steel disc with a-C:H and a-C:H:Si. The a-C:H **(Fig. 3)** coating has a fairly

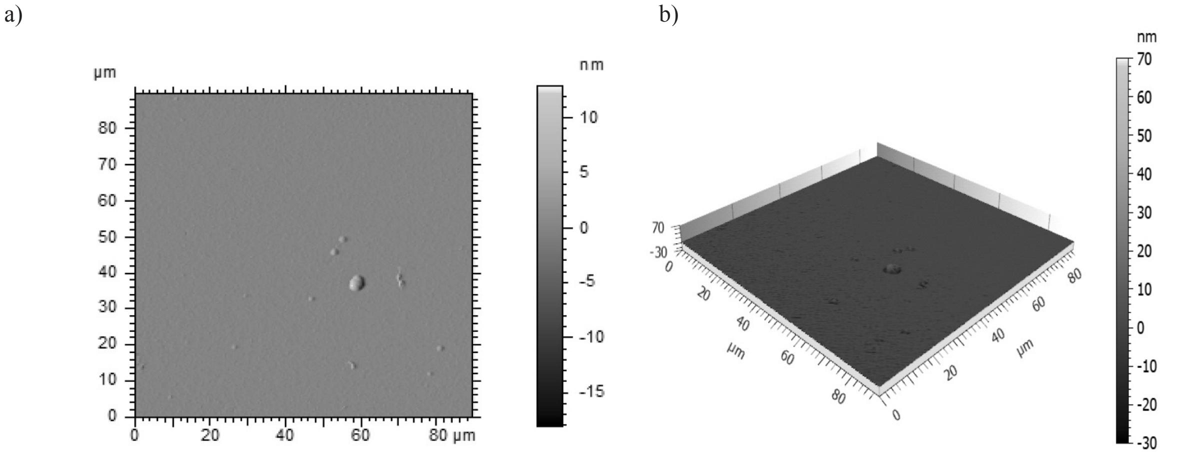


Fig. 1. Surface topography of a-C:H coating: a) 2D, b) 3D
Rys. 1. Topografia powierzchni powłoki a-C: H: a) 2D, b) 3D

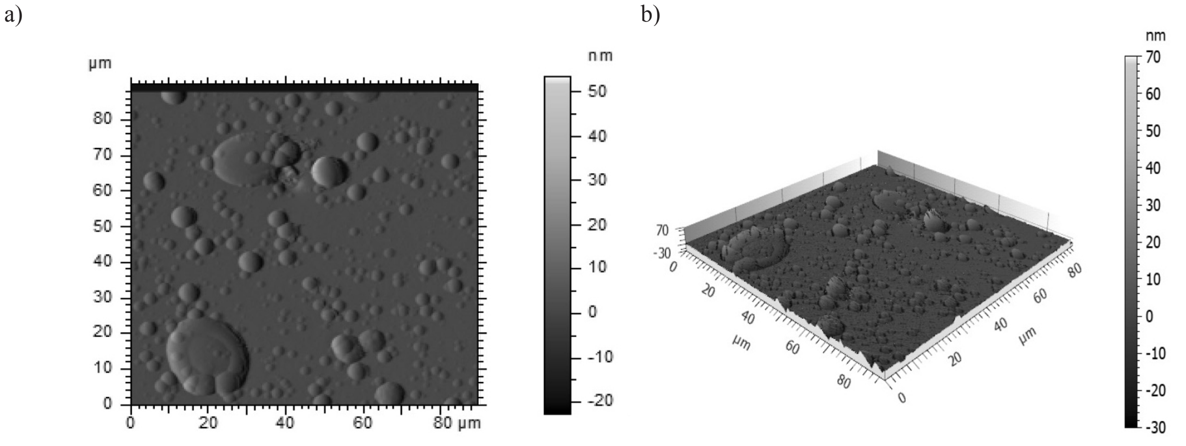
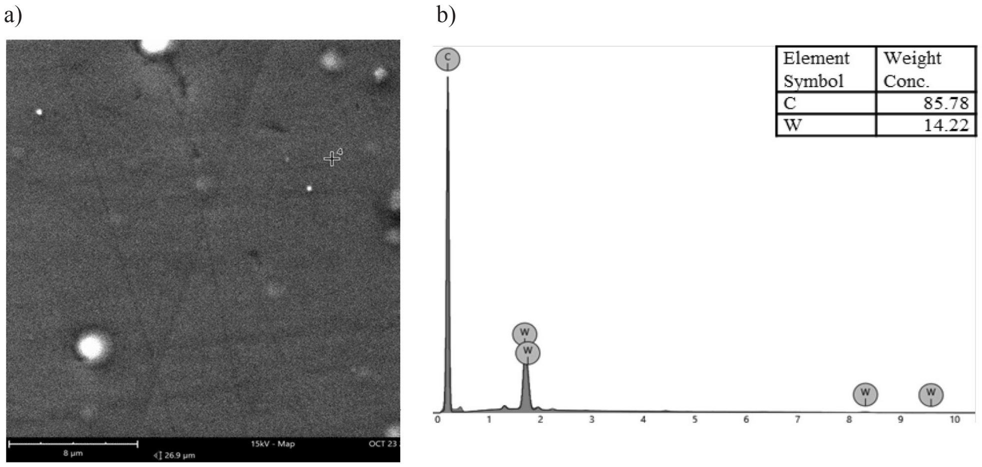
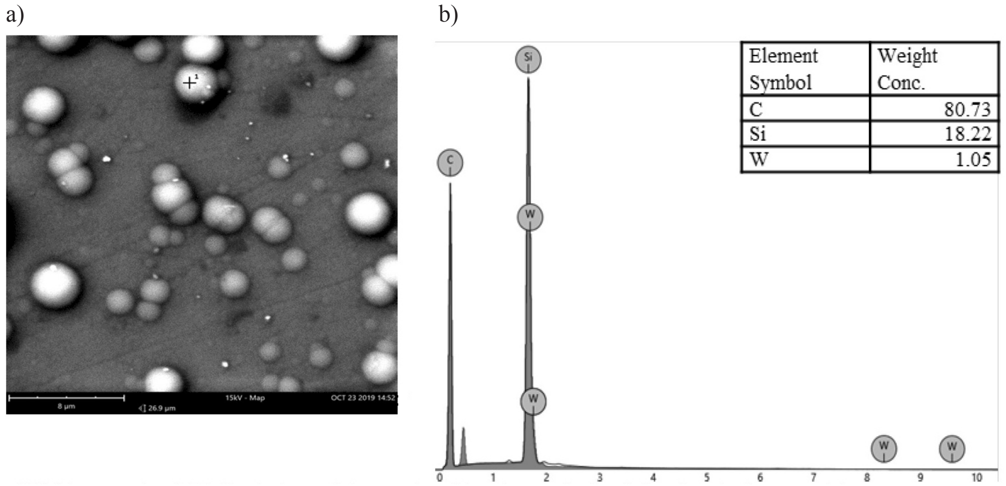


Fig. 2. Surface topography of a-C:H:Si coating: a) 2D, b) 3D
Rys. 2. Topografia powierzchni powłoki a-C: H: Si: a) 2D, b) 3D



Element Symbol	Weight Conc.
C	85.78
W	14.22

Fig. 3. SEM image of a-C:H: a) view of the coating, b) point analysis of the chemical composition
Rys. 3. Obraz SEM a-C: H: a) widok powłoki, b) punktowa analiza składu chemicznego



Element Symbol	Weight Conc.
C	80.73
Si	18.22
W	1.05

Fig. 4. SEM image of a-C:H:Si: a) view of the coating, b) point analysis of the chemical composition
Rys. 4. Tekstura powierzchni dysku stalowego 100Cr6: a) obraz izometryczny, b) profil powierzchni

homogeneous surface with band patterns that probably resulted from the preparation (grinding/polishing) of the substrate surface for coating deposition. Small quantities of dispersed particles were detected on a-C:H. Bands and dispersed particles were also present on the a-C:H:Si coating (**Fig. 4**) in greater quantities than on a-C:H. These particles were formed during deposition. In both cases, the particles consisted of carbon and tungsten. In addition, silicon was found in the selected areas of a-C: H: Si.

In the next step, surface texture was measured. **Figures 5–7** show isometric images and surface profiles of 100Cr6 steel, a-C:H, and a-C:H:Si before tribotests.

A comparison of images in **Fig. 5–7** revealed the smooth surfaces of both the uncoated and a-C:H coated steel discs. The a-C:H:Si coated surface was irregular with high hills. Analysis of the disk surface texture parameters before the tribotests (**Table 2**) showed the same roughness average represented by the Sa parameter for the substrate (steel 100Cr6) and a-C:H:Si, 0.11 µm, and 0.10 µm for the a-C:H coating. Despite similar Sa values, by far the highest peak, Sz = 5.88 µm, was observed on the a-C:H:Si surface, which is also visible on its initial profile (**Fig. 7b**). This surface also showed the highest pit represented by Sv = 4.58 µm. The substrate and the a-C:H coating had the mean square surface roughness deviation Sq = = 0.13 µm; whereas in the case of a-C:H:Si, the value of Sq was greater by over 46%. The degree of symmetry of the surface height distribution, skewness – Ssk and the measure of surface peakedness, kurtosis – Sku, provide additional information on the texture of the surfaces as these amplitude parameters are sensitive to local height variations (peaks and valleys). If the surface heights are normally distributed, Sku = 3. For 100Cr6 steel discs without and with a-C: H, the distribution was close to normal. In the case of a-C:H:Si, the value of Sku was 32.25, which indicates a small dispersion of the surface ordinate values. The Ssk parameter had positive values for all tested surfaces, which indicates that the surfaces were smooth with no deep cracks. The lowest value of the Ssk parameter was obtained for 100Cr6 steel, which was due to the presence of minor cracking on its surface.

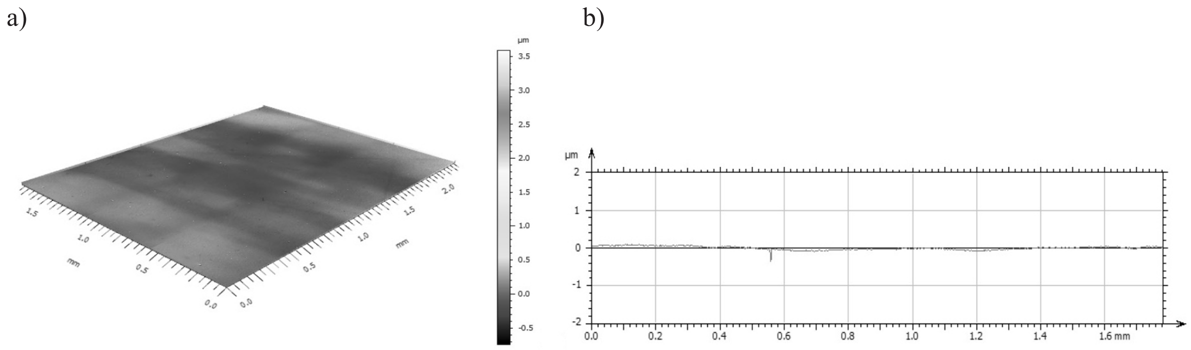


Fig. 5. Surface texture of 100Cr6 steel disk: a) isometric image, b) surface profile
Rys. 5. Tekstura powierzchni dysku stalowego 100Cr6: a) obraz izometryczny, b) profil powierzchni

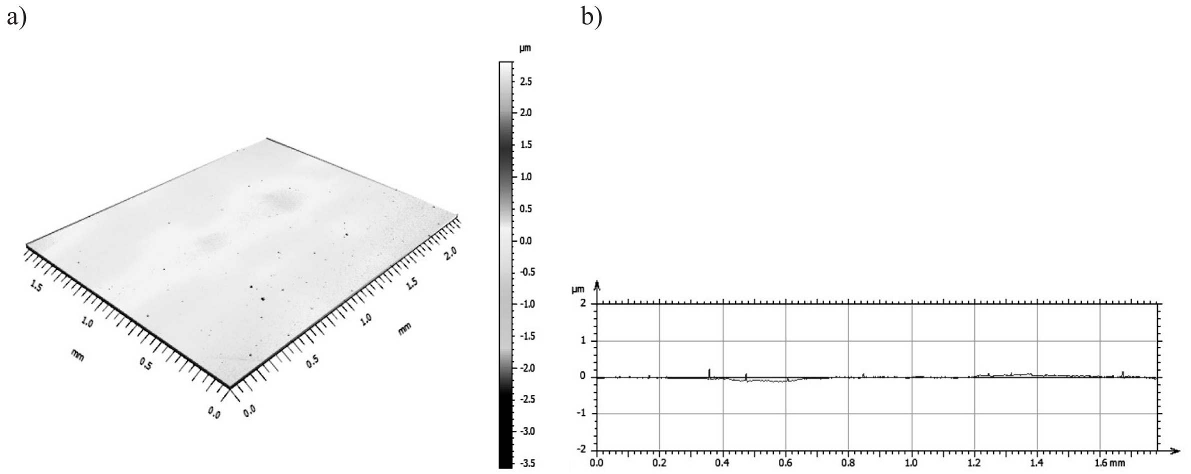


Fig. 6. Surface texture of a-C:H: a) isometric image, b) surface profile
Rys. 6. Tekstura powierzchni a-C: H: a) obraz izometryczny, b) profil powierzchni

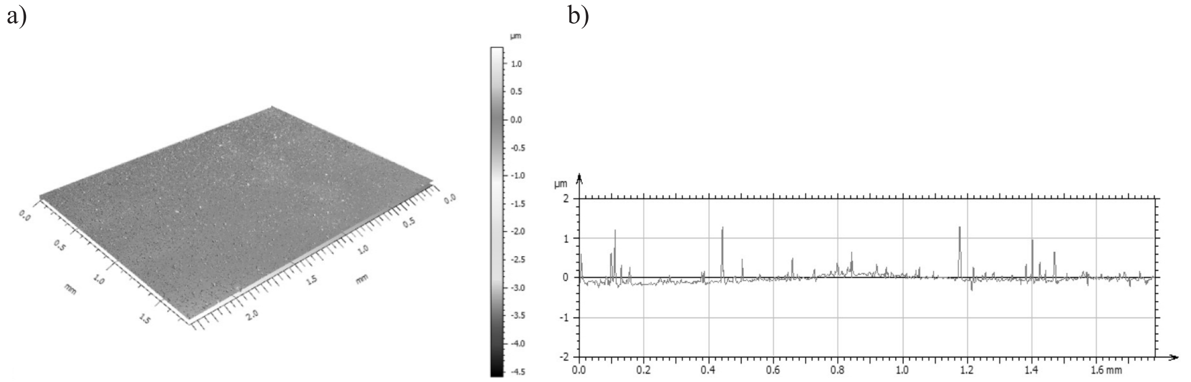


Fig. 7. Surface texture of a-C:H:Si: a) isometric image, b) surface profile
Rys. 7. Tekstura powierzchni a-C: H: Si: a) obraz izometryczny, b) profil powierzchni

Table 2. Surface texture parameters in compliance with ISO25178
Tabela 2. Parametry tekstury powierzchni zgodnie z ISO25178

Parameter	Substrate – steel 100Cr6	Coating a-C:H	Coating a-C:H:Si
Sq[µm]	0.13	0.13	0.19
Ssk	0.36	0.56	1.58
Sku	3.21	3.37	32.25
Sp[µm]	3.59	2.81	1.29
Sv [µm]	0.74	0.71	4.58
Sz[µm]	4.33	3.52	5.88
Sa[µm]	0.11	0.10	0.11

Figure 8 shows the average friction coefficients for steel 100Cr6 with and without the a-C:H and a-C:H:Si coatings, recorded for the tribo-pairs under different test conditions.

The lowest coefficient of friction, $\mu \cong 0.05$, was obtained for a-C:H in lubrication with ionic liquid, tetradecyltrihexylphosphonium bis(trifluoromethylsulfonyl) amide. The highest average coefficient of friction, $\mu \cong 0.7$, was recorded for 100Cr6 steel under dry friction. For each of the analysed substrates, the lowest friction coefficients were obtained using ionic liquid lubrication. Synthetic oil PAO-8 underperformed, and the coefficients of friction were markedly lower than those under dry friction. It is worth noting that the coefficient, μ 0.11, obtained for 100Cr6 steel with ionic liquid as a lubricant confirms its very good lubricating properties.

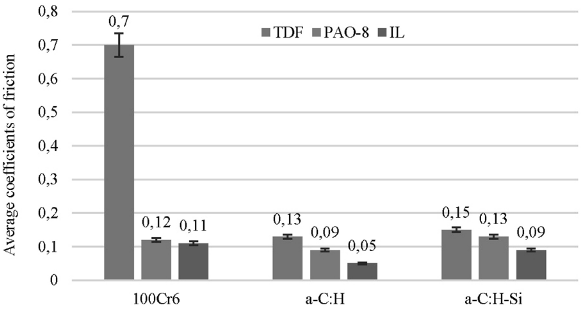


Fig. 8. Average coefficients of friction
Rys. 8. Średnie współczynniki tarcia

Figures 9–11 show the geometric structure of the surfaces after tribotests in dry friction.

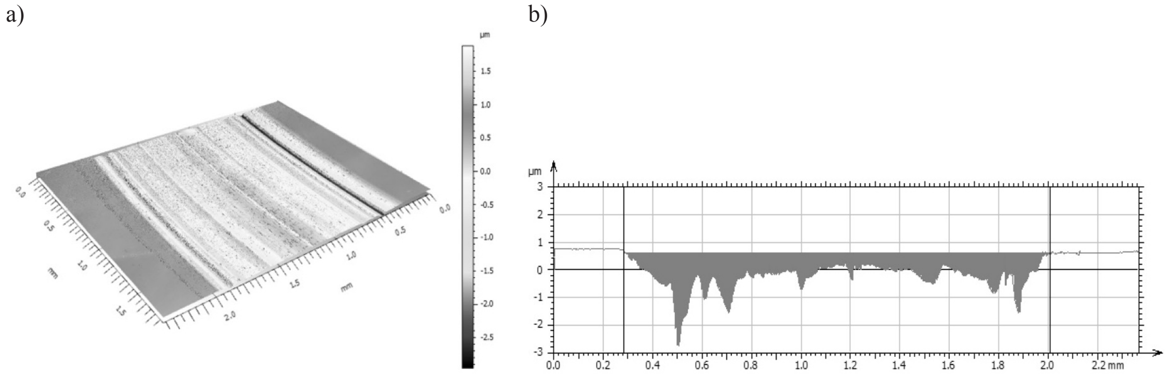


Fig. 9. Surface texture of 100Cr6 steel disk: a) isometric image, b) surface wear profile
Rys. 9. Tekstura powierzchni dysku stalowego 100Cr6: a) obraz izometryczny, b) profil zużycia powierzchni

Table 3. Surface wear profile parameters in the wear area
Tabela 3. Parametry profilu zużycia powierzchni w obszarze zużycia

Parameter	Value	Unit
Maximum depth	3.42	µm
Wear area	1600	µm^2

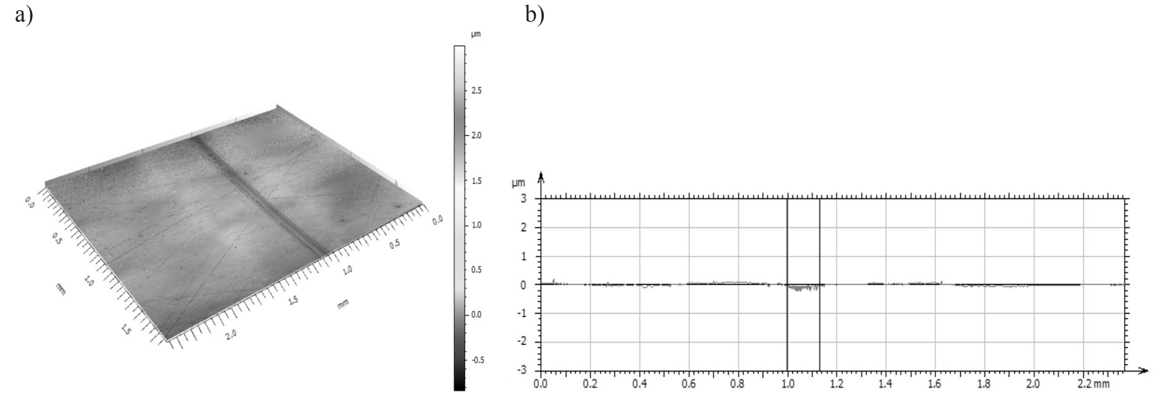


Fig. 10. Surface texture of a-C:H: a) isometric image, b) surface wear profile
Rys. 10. Tekstura powierzchni a-C: H: a) obraz izometryczny, b) profil zużycia powierzchni

Table 4. Surface wear profile parameters in the wear area
Tabela 4. Parametry profilu zużycia powierzchni w obszarze zużycia

Parameter	Value	Unit
Maximum depth	0.24	µm
Wear area	15.27	µm^2

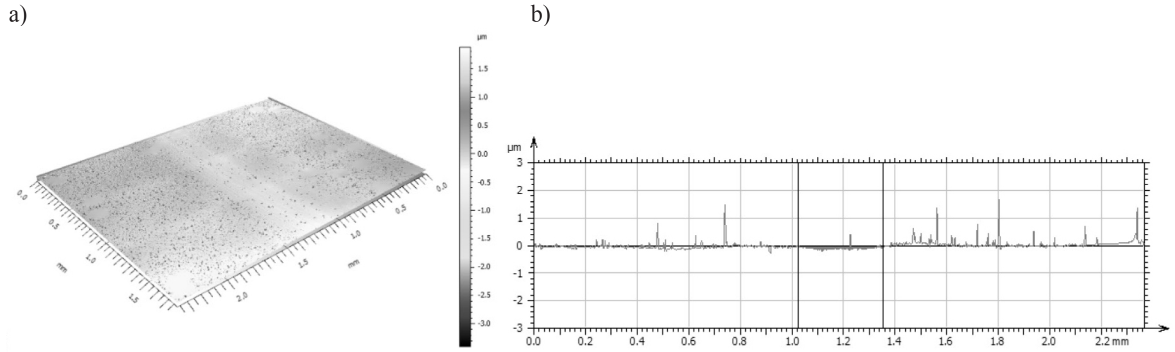


Fig. 11. Surface texture of a-C:H:Si: a) isometric image, b) surface wear profile
Rys. 11. Tekstura powierzchni a-C: H: Si: a) obraz izometryczny, b) profil zużycia powierzchni

Table 5. Surface wear profile parameters in the wear area
Tabela 5. Parametry profilu zużycia powierzchni w obszarze zużycia

Parameter	Value	Unit
Maximum depth	0.26	µm
Wear area	36.25	µm^2

Evaluation of isometric images and wear profiles after the tribotests in dry friction revealed that the largest wear area 1600 µm^2 and wear depth of 3.42 µm were recorded for steel 100Cr6. The a-C:H coating attained the smallest wear area, 15.27 µm^2, and the smallest wear depth, 0.24 µm. This is confirmed by the tribological results which show that the a-C:H coating achieved the lowest coefficient of friction, 0.13, under dry friction conditions.

Figures 12–14 show the geometric structure of the disks after tribotests in lubricated conditions with ionic liquid.

An evaluation of isometric images and surface wear profiles after the tribotests conducted in lubrication with ionic liquid revealed that largest wear area of 14.91 µm^2, and wear depths of 1.69 µm were recorded for steel 100Cr6. **Figure 14** shows no wear trace on a-C:H:Si after tribotests with ionic liquid as lubricant.

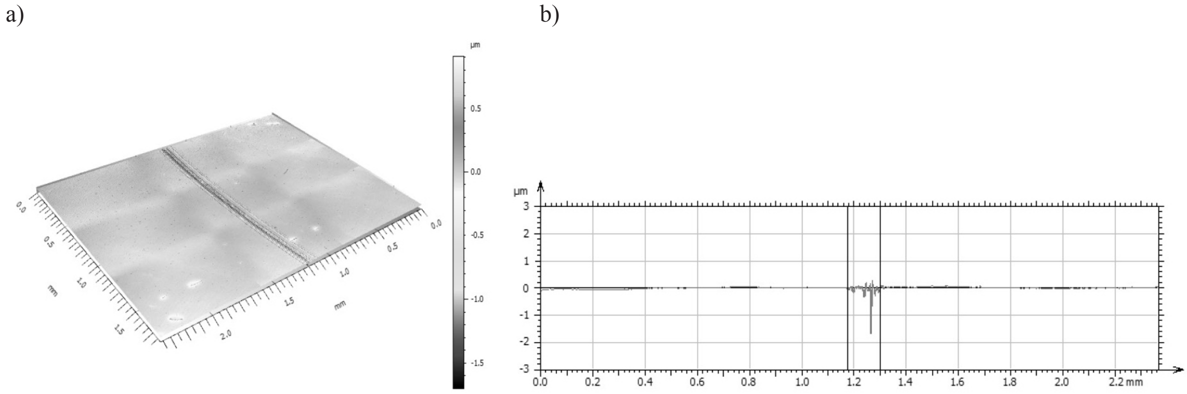


Fig. 12. Surface texture of 100Cr6 steel surface: a) isometric image, b) surface wear profile
Rys. 12. Tekstura powierzchni powierzchni stali 100Cr6: a) obraz izometryczny, b) profil zużycia powierzchni

Table 6. Surface wear profile parameters in the wear area
Tabela 6. Parametry profilu zużycia powierzchni w obszarze zużycia

Parameter	Value	Unit
Maximum depth	1.69	µm
Wear area	14.91	µm^2

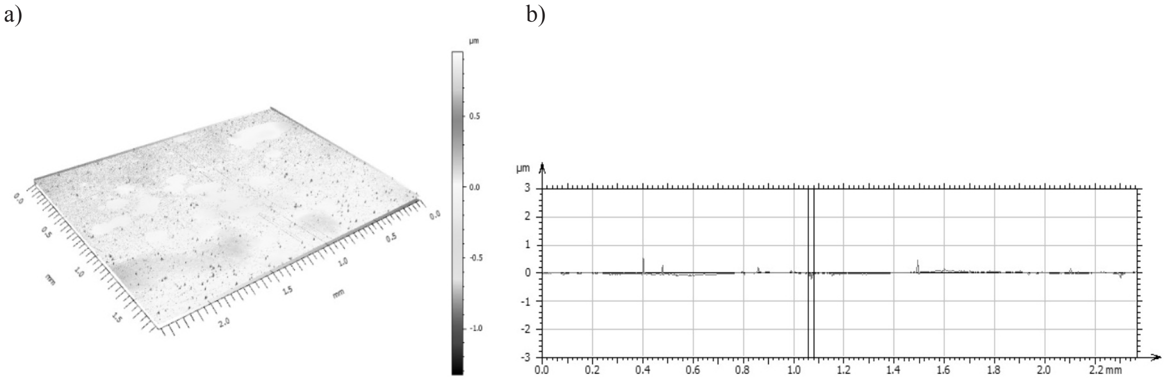


Fig. 13. Surface texture of a-C:H: a) isometric image, b) surface wear profile
Rys. 13. Tekstura powierzchni a-C: H: a) obraz izometryczny, b) profil zużycia powierzchni

Table 7. Surface wear profile parameters in the wear area
Tabela 7. Parametry profilu zużycia powierzchni w obszarze zużycia

Parameter	Value	Unit
Maximum depth	0.21	µm
Wear area	1.69	µm^2

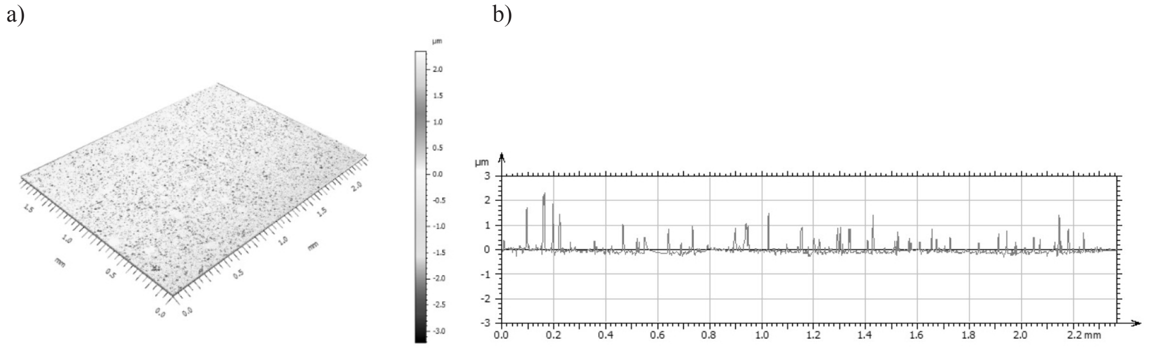


Fig. 14. Surface texture of a-C:H:Si: a) isometric image, b) surface wear profile
Rys. 14. Tekstura powierzchni a-C: H: Si: a) obraz izometryczny, b) profil zużycia powierzchni

For a-C:H, the wear area was 1.69 µm^2 and the maximum depth was 0.21 µm. These data are confirmed by the results of the tribotests, where the lowest coefficient of friction, 0.13, was obtained for a-C:H under dry friction.

Figures 15–17 show the geometric structure of the surfaces after tribotests in synthetic oil lubrication.

The largest wear trace was observed for 100Cr6 steel after the tribotests in dry friction (**Fig. 9**), with the wear depth of about 3.42 µm and the wear surface area equal to 1600 µm^2. No wear trace was observed for a-C:H:Si during tribotests in lubrication with ionic liquid or synthetic oil. On the a-C:H coating in lubrication with ionic liquid, the wear trace was about 0.21 µm. For a-C:H lubricated with synthetic oil, the wear depth was about 0.57 µm.

Table 10 compiles the texture parameters of the surfaces after tribotests to ISO 25178.

Analysis of the tests results given in **Table 10** indicates that the texture parameters obtained from the tribotests in dry friction were far better, in particular, Sa – the arithmetic mean deviation of the surface for the a-C:H coated disks (0.06 µm before the tribotests), which was 40% lower compared to the results before the tests. The high quality of surface preparation before coating deposition greatly contributed to this effect. The Sa parameter for a-C:H:Si after the tribotests in dry friction increased by 9%. Amplitude parameters sensitive to any surface variations (peaks and valleys) such as skewness Ssk and kurtosis Sku provided additional information on the texture of the surfaces under analysis. The negative values of Ssk confirm the presence of a plateau-like surface. Such values were obtained after the tribotests for 100Cr6 steel under dry friction and in lubrication with ionic liquid and synthetic oil PAO-8. The negative value

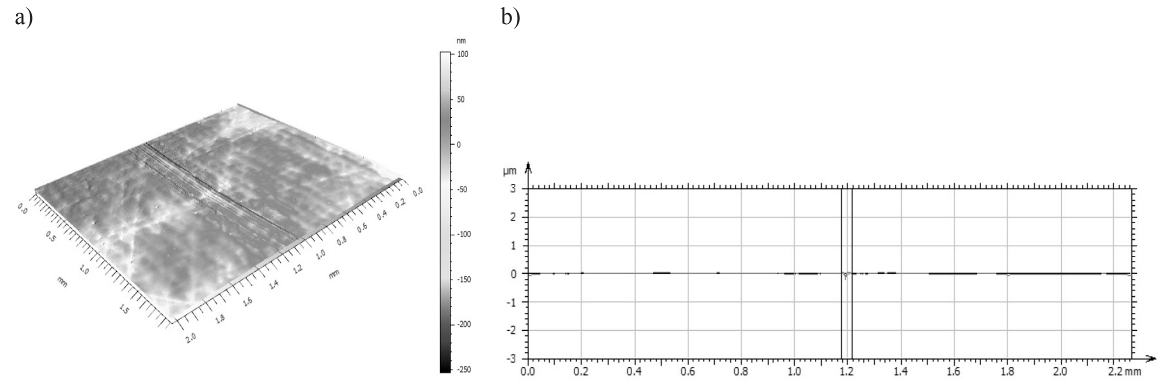


Fig. 15. Surface texture of 100Cr6 steel surface: a) isometric image, b) surface wear profile

Rys. 15. Tekstura powierzchni powierzchni stali 100Cr6: a) obraz izometryczny, b) profil zużycia powierzchni

Table 8. Surface wear profile parameters in the wear area

Tabela 8. Parametry profilu zużycia powierzchni w obszarze zużycia

Parameter	Value	Unit
Maximum depth	0,20	µm
Wear area	0,77	µm^2

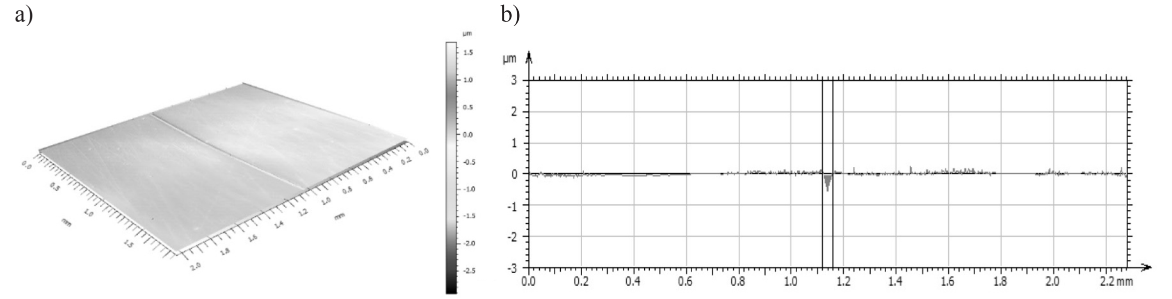


Fig. 16. Surface texture of a-C:H: a) isometric image, b) surface wear profile

Rys. 16. Tekstura powierzchni a-C: H: a) obraz izometryczny, b) profil zużycia powierzchni

Table 9. Surface wear profile parameters in the wear area

Tabela 9. Parametry profilu zużycia powierzchni w obszarze zużycia

Parameter	Value	Unit
Maximum depth	0,57	µm
Wear area	8,41	µm^2

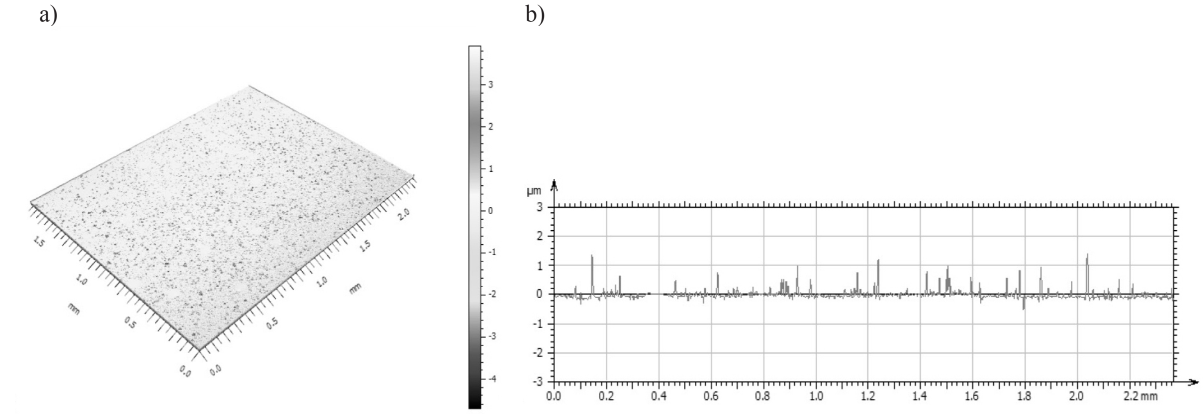


Fig. 17. Surface texture of a-C:H:Si: a) isometric image, b) surface wear profile

Rys. 17. Tekstura powierzchni a-C: H: Si: a) obraz izometryczny, b) profil zużycia powierzchni

Table 10. Texture parameters of the samples after tribotests
Tabela 10. Parametry tekstury próbek po testach plemiennych

Substrate and lubrication type	**Parameters**						
	Sp [µm]	Sv [µm]	Sz [µm]	Sa [µm]	Sq [µm]	Ssk	Sku
100Cr6 *TTS*	1.88	2.69	4.84	0.45	0.50	-1.17	5.31
a-C:H *TTS*	3.00	0.84	3.84	0.06	0.07	0.17	21.01
a-C:H:Si *TTS*	1.88	3.40	5.28	0.12	0.19	1.08	35.67
100Cr6 *IL*	0.91	1.70	2.61	0.04	0.09	-10.67	184. 8
a-C:H *IL*	0.95	1.32	2.28	0.05	0.07	2.53	35.34
a-C:H:Si *IL*	2.35	3.22	5.56	0.12	0.25	3.70	37.47
100Cr6 *PAO-8*	0.10	0.27	0.37	0.07	0.02	-0.001	0.006
a-C:H *PAO-8*	1.70	2.91	4.61	0.06	0.09	-3.28	35.86
a-C:H:Si *PAO-8*	3.91	4.70	8.62	0.15	0.31	4.09	44.38

of Ssk was also obtained for a-C:H after tribotests with PAO-8 lubrication. The high values of Sku, mainly in the case of a-C:H:Si, but also a-C:H and steel 100Cr6 in lubrication with ionic liquid, confirm high slenderness of the ordinate distribution. The highest values of Sp, Sv, and Sz parameters were recorded for the a-C:H:Si coating.

CONCLUSIONS

Surface topography results indicated a good quality of the a-C:H coating. The isometric views and topography images of a-C:H:Si showed evident dispersed inclusions from silicon, as confirmed by the EDS analysis of its chemical composition.

During the tribological tests, the lowest average coefficient of friction in lubrication with ionic liquid, $\mu \cong 0.05$, was obtained for a-C:H. A coefficient twice as high, $\mu \cong 0.11$, was obtained for a-C:H:Si. The coefficient of friction for the 100Cr6 steel disk was more than 500% higher, $\mu \cong 0.7$. Markedly lower coefficients for a-C:H and a-C:H:Si in dry friction were the result of the self-lubricating properties – the presence of graphite in the DLC coating. The use of the PAO-8 synthetic oil significantly reduced the coefficient of friction. The coefficient for 100Cr6 steel in lubrication with ionic liquid, $\mu \cong 0.11$, confirmed good lubricated properties of trihexyltetradecylphosphonium bis(trifluoromethylsulfonyl) amide.

Observations of the disk wear after the tribotests showed evident reduction in wear in the case of a-C:H and a-C:H:Si coated samples. Analysis of the isometric views and wear profiles after the tribotests in lubrication with ionic liquid found no wear trace on a-C:H:Si. The lowest level of wear, about 0.21 µm, was found for a-C:H. The deepest wear, about 3.42 µm, was found in the 100Cr6 steel disk in dry friction.

Diamond-like carbon coatings a-C:H and a-C:H:Si lubricated with trihexyltetradecylphosphonium bis(trifluoromethylsulfonyl) amide ionic liquid both contribute to the reduction of friction coefficient and decrease the wear of the samples tested.

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