ISSN 0208-7774 TRIBOLOGIA 6/2018 p. 97–106

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THE INFLUENCE OF SILICON-DOPED DIAMOND-LIKE CARBON COATING ON THE WEAR OF IONIC LIQUID LUBRICATED FRICTION PAIRS

WPŁYW POWŁOKI DIAMENTOPODOBNEJ DOMIESZKOWANEJ KRZEMEM NA ZUŻYCIE WĘZŁÓW TARCIA SMAROWANYCH CIECZĄ JONOWĄ

Key words: DLC coating, 100Cr6 steel, ionic liquid, friction, wear.

Abstract This article reports the results of the study of an a-C:H:Si coating doped with silicon and produced by chemical deposition (PACVD). The effect of the coating on the tribological behaviour of IL-lubricated friction pairs was evaluated. The properties of the 100Cr6 steel specimens with and without the coating were compared. Scanning electron microscopy (SEM) and energy dispersive X-ray (EDX) analysis were used for imaging the morphology of the coating surfaces and cross-sections and for identifying the elements in the coating composition. The contact angle of the investigated surfaces was measured with an optical tensiometer. Friction tests were performed on a ball-on-disc tribometer under dry friction and when lubricated with trihexyltetradecylphosphonium bis(trifluoromethylsulfonyl) amide ionic liquid. The geometrical structure of the surfaces before and after the tribological tests was measured using an optical profilometer. The ionic liquid used with the silicon-doped diamond-like coating under friction conditions reduced the coefficient of friction and wear. The results obtained from the tests and analysis allow for the conclusion that the use of DLC coatings a-C:H:Si lubricated with trihexyltetradecylphosphonium Bis(trifluoromethylsulfonyl) amide contributes towards the improvement of tribological properties of sliding surfaces under friction.

Słowa kluczowe: powłoka diamentopodobna, stal 100Cr6, ciecz jonowa, tarcie, zużycie.

Streszczenie Artykuł poświęcony jest badaniom powłoki diamentopodobnej domieszkowanej krzemem typu a-C:H:Si uzyskanej w procesie chemicznego osadzania z fazy gazowej wspomaganego plazmą PACVD. Ocenie poddano wpływ powłoki na właściwości tribologiczne węzłów tarcia smarowanych cieczą jonową (Ionic Liquid) IL. W analizie porównano próbki ze stali 100Cr6 bez powłoki oraz pokryte powłoką diamentopodobną. Przy użyciu skaningowego mikroskopu elektronowego SEM (Scanning Electron Microscopy) wyposażonego w mikroanalizator rentgenowski EDS (X-ray Energy Dispersive Spectroscopy), zobrazowano morfologię powierzchni powłok, ich przekroje poprzeczne oraz dokonano identyfikacji pierwiastków wchodzących w skład powłoki. Wykonano również pomiary kąta zwilżania badanych powierzchni na optycznym tensjometrze. Badania tarciowe przeprowadzono na tribotesterze pracującym w skojarzeniu trącym kula–tarcza w warunkach tarcia technicznie suchego oraz z zastosowaniem smarowania cieczą jonową triheksyltetradecylphosphonium bis(triffluoromethylsulfonyl) amide. Charakterystykę struktury geometrycznej próbek przed oraz po badaniach tribologicznych przeprowadzono na profilometrze optycznym. Wyniki badań wskazały, że użyta ciecz jonowa w skojarzeniu trącym z powłoką diamentopodobną domieszkowaną krzemem wpłynęła na zmniejszenie współczynnika tarcia oraz zużycia. Otrzymane wyniki wskazują, że zastosowanie powłok diamentopodobnych typu a-C:H:Si smarowanych triheksyltetradecylphosphonium bis(triffluoromethylsulfonyl) amidem sprzyja poprawie właściwości tribologicznych węzłów tarcia w ruchu ślizgowym.

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INTRODUCTION

Adverse effects of friction are often mitigated by applying coatings of various types. Diamond-like carbon (DLC) coatings raise a lot of interest due to their unique tribological, mechanical, optical, corrosive, and biocompatible properties [**L. 1–3**]. For this reason the coating can be used in many industries in automotive, tool, electronic, food, and medical sectors. The popularity of DLC coatings is steadily growing in engineering applications. They are increasingly used in lubricated tribosystems and at high loads under boundary or mixed lubrication [**L. 2, 4**]. DLC application reports found in the literature also include the use of the coatings for elements of implants and medical prostheses in orthopaedics and cardiac surgery [**L. 5–7**].

DLC coatings can be deposited using a number of techniques, as described elsewhere. Generally, two processes are used: physical deposition (PVD) and chemical vapour deposition (CVD). These processes are often enhanced with plasma for lowering the deposition temperature [**L. 1, 8**]. DLC coating properties are defined by the method and parameters of deposition, structure, and doping with metals or non-metals [**L. 3**]. Tribological characteristics of the coatings are good both under dry friction and under boundary friction. A number of factors affect tribological properties under boundary friction. These are the elements being added to the coating, base oil type, chemical additives and their concentrations, physico-chemical parameters of lubricants, contact pressure, sliding velocity, temperature, etc. [**L. 3, 4, 9–11**].

There has been a rapid increase in the use of low-temperature ionic liquids (LTILs) as lubricants. Ionic liquids are organic substances that have a normal melting point lower than the boiling point of water. Salts that melt at temperatures lower than room temperature (25°C) are a special group of ILs. Ionic liquids are composed of a large, asymmetric cation and a weakly-coordinating anion that can be either organic or inorganic [**L. 12–16, 23, 24**]. In Poland, the pioneering studies of ionic liquids as lubricants were conducted by the team from T. Kaldoński Military University of Technology [**L. 12, 13]** and the team led by M. Sułek at the Radom University of Technology **[L. 14–16]**.

Since they do not release volatile organic compounds, ILs are considered to be "green lubricants" of unique properties such as high stability and thermo-oxidative durability, flammability, high ionic conductivity, and unlimited miscibility with organic compounds. A strong electrostatic bonding in their structure improves their lubricating properties. These parameters ensure that ionic liquids meet all the requirements for high-performance lubricants [**L. 21, 24, 25**]. Some of the liquids have better tribological properties than the commonly used synthetic and mineral oils. The application of ionic liquids instead of hydrocarbon base oils can substantially reduce the emissions to the atmosphere of by-products resulting from the release of sulphur dioxide, nitrogen oxides, and other toxic chemicals. Many studies have focused on tribological applications of imidazole liquids containing tetrafluoroborate (BF4) and hexafluorophosphate (PF6) anions, characterized by both low friction and anti-wear behaviour [**L. 15–18, 21–24**].

The aim of this study was to evaluate the effect of the DLC coating on the wear of the ionic liquid-lubricated friction pair. The subject matter discussed in this article results from the current problems related to the use of materials with specific functional properties, including low-friction DLC coatings and sustainable lubricants compliant with the binding legal regulations and occupational health and safety rules. This study uses the sulphur and phosphorus-based trihexyltetradecylphosphonium Bis(trifluoromethylsulfonyl) amide ionic liquid, characterized by anion hydrophobicity and hydrolytic stability.

MATERIALS AND METHODS

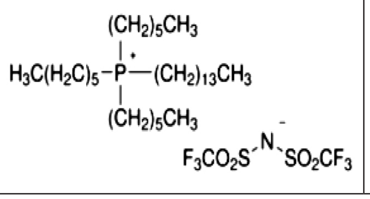
The coating under analysis was a silicon-doped diamond-like coating a-C:H:Si deposited by plasma assisted chemical vapour deposition (PACVD). The substrate for the coating was bearing steel 100Cr6, which has the chemical composition summarized in **Table 1**.

Table 1. Chemical composition of 100Cr6 steel
Tabela 1. Skład chemiczny stali 100Cr6

Element	**C**	**Mn**	**Si**	**P**	**S**	**Cr**
Percentage, %	0.93– 1.05	0.25– 0.45	0.15–0.35	< 0.025	< 0.030	1.35–1.60

Trihexyltetradecylphosphonium bis(trifluoromethylsulfonyl) amide ionic liquid with very good tribological properties was used in the tribological tests. The structure and selected properties of the IL under test are shown in **Table 2**.

Table 2. Structure and selected properties of the ionic liquid (IL)
Tabela 2. Budowa i wybrane właściwości cieczy jonowej IL

Name	Molecular formula	Graphic formula	Molecular weight [g/mol]	Density [g/cm^3]	Melting point [ºC]
Trihexyltetra-decylphosphonium bis(trifluorome-thylsulfonyl) amide	$[CH_3(CH_2)_5]_3$ $P[N(SO_2CF_3)_2]$ $(CH_2)_{13}CH$		764	1.07	-72.4

The aim of the research was to assess the tribological properties of the diamond-like coating doped with silicon under friction conditions, lubricated with the selected ionic liquid.

The elements constituting the a-C:H:Si coating were identified using a JSM-7100F scanning electron microscope with an EDS microanalyser.

Wetting is a process that takes place in three-phase systems in which a given liquid displaces gas or other liquid from the surface of a liquid or solid, and the contact angle is its measure for solids. The most popular methods of determining free surface energy include the Wu and Zisman method. The contact angles of the surface of the tested 100Cr6 steel specimens without and with the a-C: H: Si coating were measured with a Attension Theta tensiometer manufactured by Biolin Scientific (**Figure 1**) that allows determining the contact angle from the analysis of drop shape.

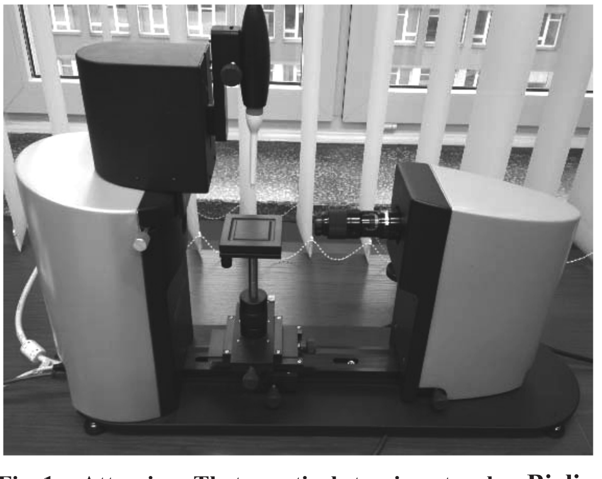


Fig. 1. Attension Theta optical tensiometer by Biolin Scientific
Rys. 1. Widok ogólny tensjometru Attension Theta Biolin Scientific

Geometry analysis of the SGP surface of the elements before and after the tribological tests was performed using a Talysurf CCI Lite optical profilometer.

Tribological tests were carried out on the ball-on-disc Anton Paar TRB tribometer (**Figure 2**).

Fig. 2. Anton Paar TRB tribometer
Rys. 2. Widok ogólny tribotestera TRB Anton Paar

The test parameters were as follows:

- Friction pair: a ball 6 mm in diameter, made of 100Cr6 steel – a 100Cr6 steel disc without coating and coated with the DLC (a-C:H:Si);
- Load: P = 50 N;
- Sliding speed: v = 0.1 m/s;
- Sliding distance: s = 1000 m;
- Humidity: 45 ± 1%; and,
- Ambient temperature: T_0 = 24±1˚C.

The tests were performed under dry conditions and with IL lubrication – trihexyltetradecylphosphonium bis(trifluoromethylsulfonyl) amide.

RESULTS AND DISCUSSION

Figure 3 shows a SEM image of the a-C:H:Si coating surface morphology with its characteristic X-ray spectra. The observations revealed the inhomogeneous surface of the coating **(Figure 3a)**. A scanning electron microscopy image of the coating cross-section and the measured thickness of individual layers are shown in **Figure 4**, and the coating cross-section with the linear analysis of the elemental distribution is shown in **Figure 5.**

Point analysis of the chemical composition indicates the inclusions of dispersed silicon particles on the surface of the DLC coating, as visible at Point 1 (**Figure 3b**). The analysis also revealed a peak from oxygen, most likely formed as a result of the oxidation of the silicon-rich surface. The homogeneous surface of the coating, characterized by Point 2, consists mainly of carbon and tungsten atoms, which are part of the components necessary to form the coating. (**Figure 3c**). At both points of the analysis, the presence of chromium was found, originating from the interlayer applied to improve the adhesion between the DLC coating and the steel substrate.

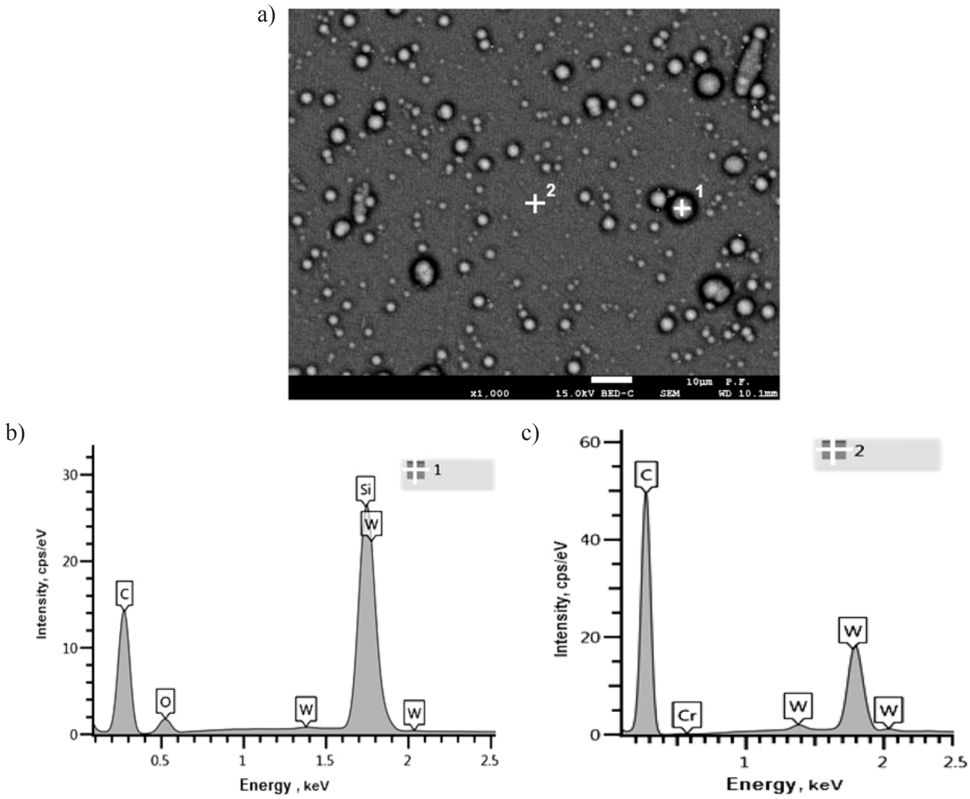


Fig. 3. SEM image: a) a-C:H:Si coating morphology and characteristic X-ray spectra for the a-C:H:Si coating at, b) Point 1, c) Point 2

Rys. 3. Obraz SEM: a) morfologia powierzchni powłoki a-C:H:Si oraz widma charakterystycznego promieniowania rentgenowskiego powłoki a-C:H:Si w: b) punkcie 1, c) punkcie 2

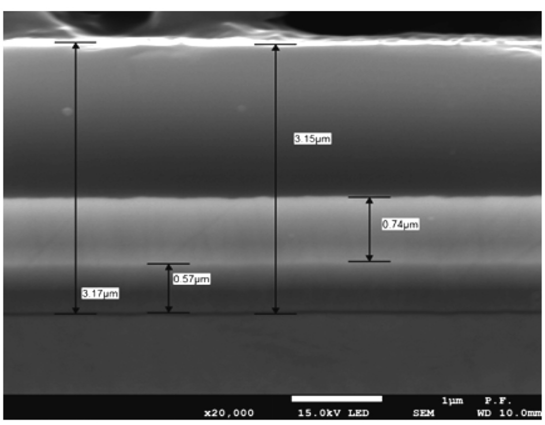


Fig. 4. SEM image: a-C:H:Si coating cross-section and measured thickness

Rys. 4. Obraz SEM: przekrój poprzeczny powłoki a-C:H:Si wraz z pomiarem jej grubości

Elemental distribution analysis (**Figure 5**) confirmed that the composition of the coating was consistent with the composition assumed during the manufacturing process. The top layer of the coating with a thickness of approx. 1.86 µm was mainly carbon and silicon. The interlayer with a thickness of approx. 0.74 µm was composed mainly of tungsten, and another interlayer with a thickness of approx. 0.57 µm consisted of chromium atoms. The use of chromium and tungsten interlayers aimed at increasing the adhesion of the diamond-like coating to the substrate. The total coating thickness was about 3.17 µm.

The next stage of the research included measurements of the contact angle. Drops of the ionic liquid – trihexyltetradecylphosphonium bis (trifluoromethylsulfonyl) amide, distilled water, and glycerine were applied to the surface of the test

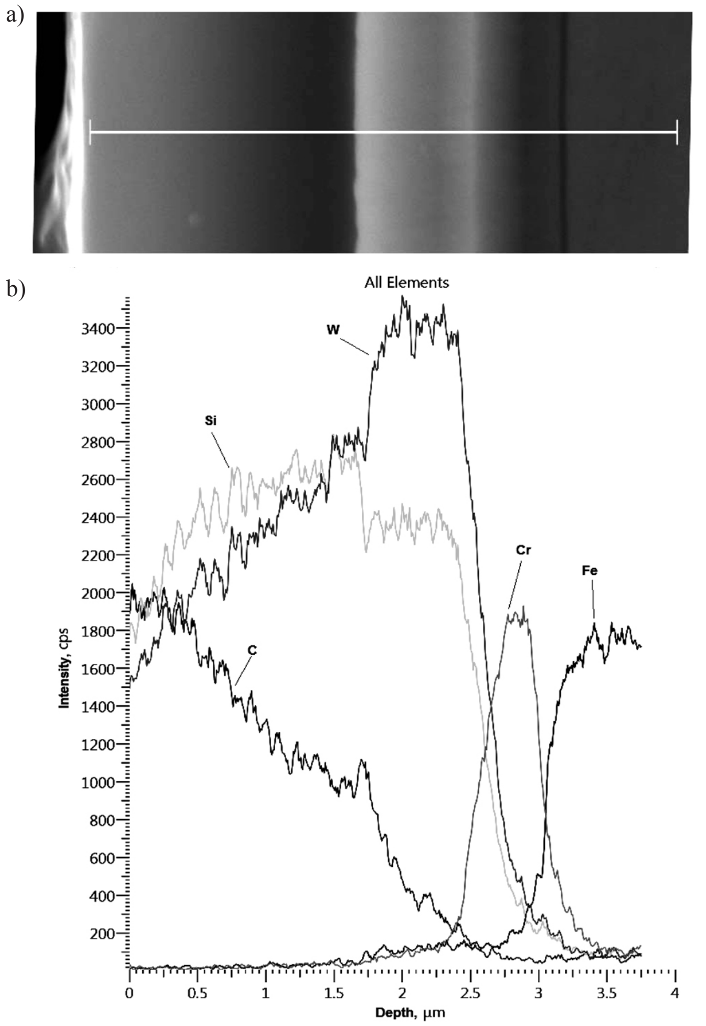


Fig. 5. SEM image: a) a-C:H:Si coating cross-section, b) linear distribution of chemical elements
Rys. 5. Obraz SEM: a) przekrój poprzeczny powłoki a-C:H:Si, b) liniowy rozkład pierwiastków

specimens using a 1 ml centrifugation syringe equipped with a chromatography needle. Contact angle readings were done after 10 s from the time the drop was placed. The mean value read from both sides of the drop was taken as the measured value of the contact angle. The measurement was carried out in three series of 20 repetitions. **Figure 6** shows sample photos taken during the measurement of the contact angle of the 100Cr6 steel disc and the disc with the a-C: H: Si coating lubricated with ionic liquid.

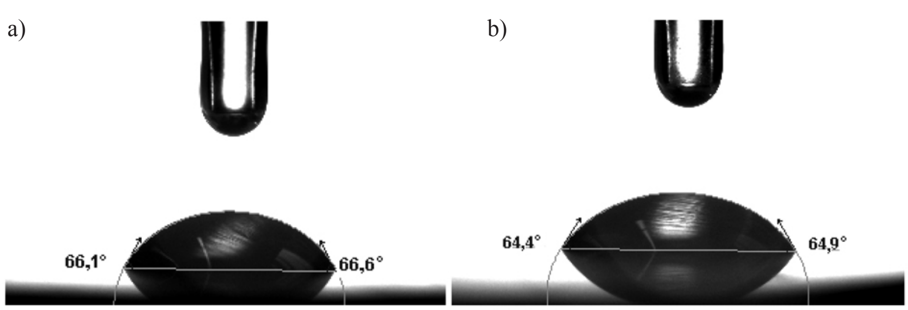


Fig. 6. Sample images of the ionic liquid drop on the surface: a) of 100Cr6 steel, b) a-C:H:Si coating
Rys. 6. Przykładowe obrazy kropli cieczy jonowej na powierzchni: a) stali 100Cr6, b) powłoki a-C:H:Si

Average contact angles of the surfaces of 100Cr6 disc and with the a-C:H:Si coating are summarized in **Figure 7**. The contact angle measurements were performed using the trihexyltetradecylphosphonium bis(trifluoromethylsulfonyl) amide ionic liquid, distilled water, and glycerine.

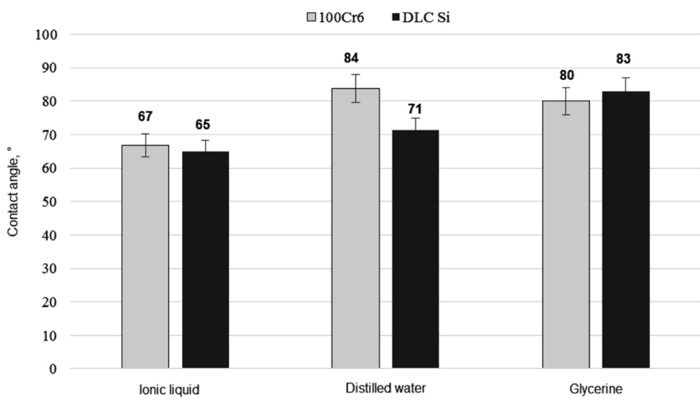


Fig. 7. Contact angle against surfaces types and liquids being tested
Rys. 7. Wykres zależności kąta zwilżania od rodzaju powierzchni i badanych cieczy

The smallest contact angle of 65° was obtained for the ionic liquid on the surface of 100Cr6 steel with a-C:H: Si coating. For the ionic liquid on the surface of steel 100Cr6 without the coating, the contact angle was 67°. The recorded values of trihexyltetradecylphosphonium bis(trifluoromethylsulfonyl) amide contact angles were significantly lower for both of the above-mentioned surfaces compared to the distilled water and glycerine contact angles.

The geometric structures of the disc and coating surfaces were measured. **Figures 8** and **9** show isometric images and primary surface profiles of 100Cr6 steel and the a-C:H:Si coating, respectively.

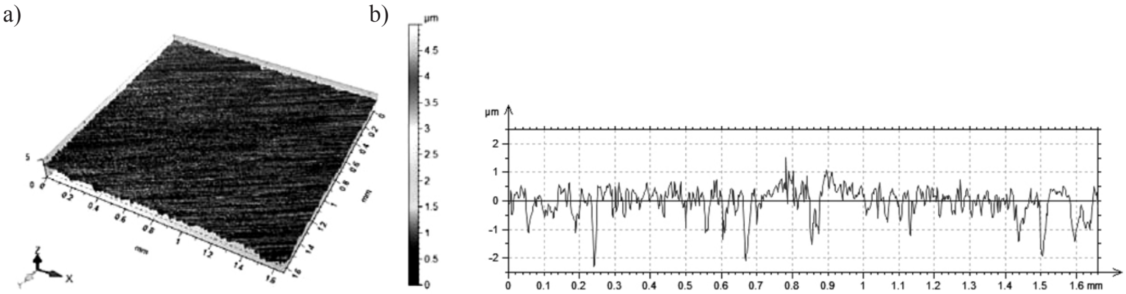


Fig. 8. Geometric structure of the 100Cr6 disc: a) isometric image, b) primary profile of the surface before testing
Rys. 8. Struktura geometryczna powierzchni tarczy ze stali 100Cr6: a) obraz izometryczny, b) profil pierwotny powierzchni przed testami tribologicznymi

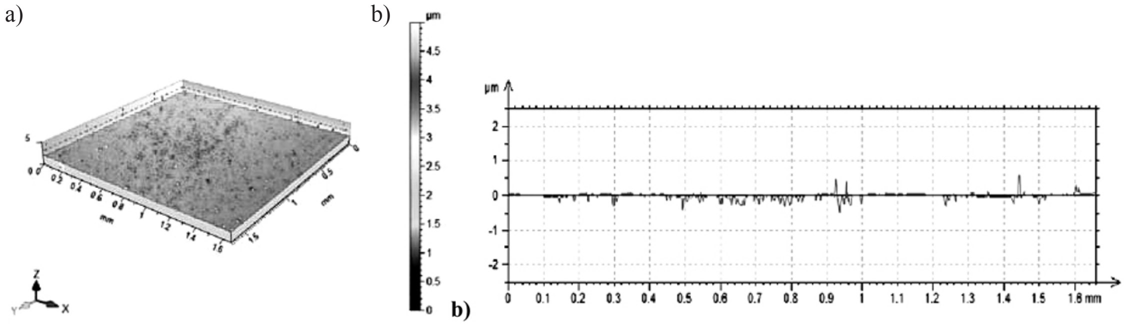


Fig. 9. Geometric structure of a-C:H:Si: a) isometric image, b) primary profile of the surface before tribological testing
Rys. 9. Struktura geometryczna powierzchni powłoki a-C:H:Si: a) obraz izometryczny, b) profil pierwotny powierzchni przed testami tribologiczymi

Analysis of the data in **Figure 8** indicates that the disc surface with the a-C:H:Si coating was very smooth, with average peaks of 0.05 µm. One valley of 0.2 µm was observed, which probably resulted from the specimen preparation process prior to coating deposition.

Figure 10 shows average friction coefficients for the 100Cr6 steel without and with the a-C:H:Si coating, recorded during dry and IL lubricated friction.

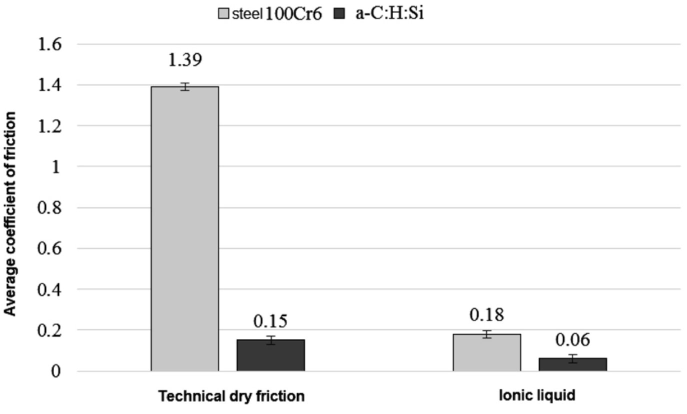


Fig. 10. Average coefficients of friction
Rys. 10. Średnie współczynniki tarcia

The lowest average friction coefficient was obtained for a-C:H:Si $\mu \cong 0.06$. The highest average coefficient was recorded for 100Cr6 without the coating and for the lubricant $\mu \cong 1.53$. The a-C:H:Si coating under dry friction had a noticeably lower friction coefficient than the 100Cr6 steel lubricated with ionic liquid $\mu \cong 0.15$.

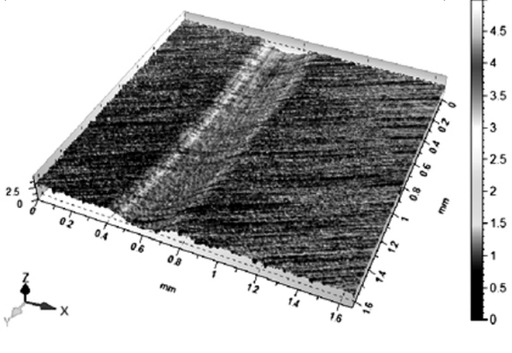
Figures 10 and **11** show the geometric structures of the 100Cr6 specimen without and with the a-C:H:Si coating after tribological tests using ionic liquids.

The wear scar that occurred during the tribological tests, **Figure 11,** was about 2.36 µm deep, and its surface area was 466.26 µm^2.

In order to compare the results for 100Cr6 steel (**Figure 11**), **Figures 12a** and **b** uses the same scale as for the steel. Since no wear scar was observed in **Figures 12a** and **b,** to better illustrate the wear area, the geometric structure analysis of the surface was performed for ranges 0–1 µm (**Figure 12c**) and –0.5–0.5 (**Figure 12d**).

No evidence of wear scar was observed on the specimens with the a-C:H:Si coating after tribological tests with ionic liquid lubrication. A more detailed analysis presented in **Figures 12c and d** revealed peaks in the friction zone, as illustrated by the primary profile. The above change may be the result of the formation of a permanent lubricating film resulting from the interaction of the ionic liquid with the a-C:H:Si coating. This requires further research and analysis that will be implemented in the future.

a) b)



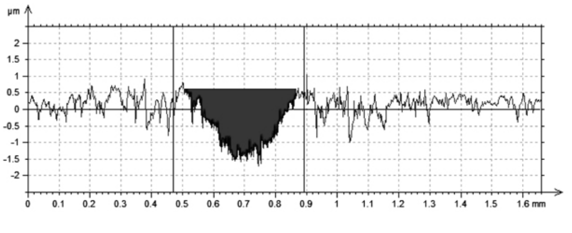


Fig. 11. Geometric structure of the 100Cr6 surface: a) isometric image, b) primary profile of the surface after tribological testing with ionic liquid lubrication
Rys. 11. Struktura geometryczna powierzchni ze stali 100Cr6: a) obraz izometryczny, b) profil pierwotny powierzchni po testach tribologicznych ze smarowaniem cieczą jonową

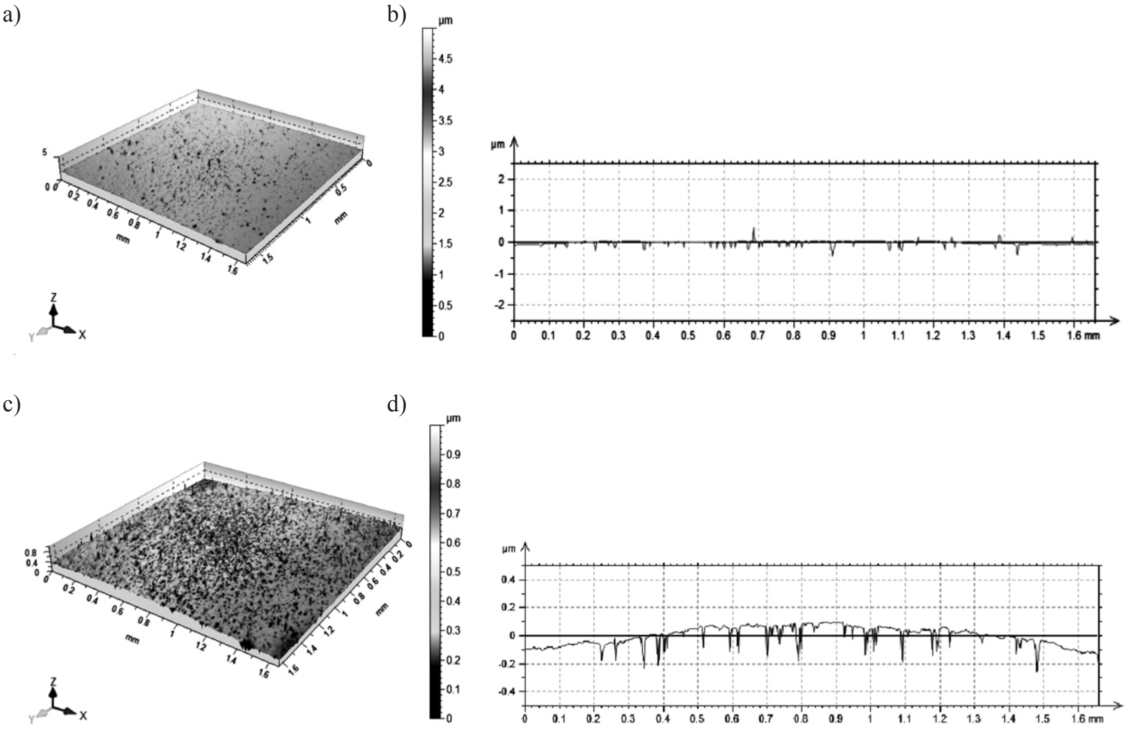


Fig. 12. Geometric structure of a-C:H:Si: a), c) isometric image, b), d) primary profile of the surface after tribological testing with ionic liquid lubrication

Rys. 12. Struktura geometryczna powierzchni powłoki a-C:H:Si: a), c) obrazy izometryczne, b), d) profile pierwotne po testach tribologicznych ze smarowaniem cieczą jonową

The above findings support the statement that the use of trihexyltetradecylphosphonium bis (trifluoromethylsulfonyl) amide as lubricant for the silicon doped DLC clearly improved the tribological properties of the friction pair being analysed.

Table 3 compiles geometric structure parameters for the surfaces analysed according to ISO 4287.

Analysis of the data in **Table 3** demonstrates that the lowest arithmetic average value of surface roughness grades, *Ra* = 0.02 μm, were obtained from the a-C:H:Si coating after the tribological tests. This value decreased by 50% as compared to the results obtained before the testing. Likewise, *Ra* after tribological tests on the 100Cr6 steel substrate decreased by more than 38% relative to the value of *Ra* before the tests. After tribological tests on the a-C:H:Si coating, the mean height of profile irregularities, *Rc*, was the lowest relative to the *Rc* value before the tests, with about 43%

Table 3. Parameters of the specimen geometric structure as per ISO 4287

Tabela 3. Parametry struktury geometrycznej badanych próbek według ISO 4287

Geometric structure parameters	Substrate – 100Cr6 steel before tribotesting	Substrate – 100Cr6 steel after tribotesting	a-C:H:Si coating before tribotesting	a-C:H:Si coating after tribotesting
Rp [μm]	0.71	0.53	0.21	0.03
Rv [μm]	1.48	0.74	0.25	0.18
Rz [μm]	2.18	1.28	0.47	0.22
Rc [μm]	1.04	0.57	0.21	0.12
Rt [μm]	3.07	1.63	0.98	0.26
Ra [μm]	0.31	0.19	0.04	0.02
Rq [μm]	0.42	0.23	0.07	0.04
Rsk	–1.01	–0.58	–1.41	–2.98
Rku	4.57	3.45	17.02	13.56

reduction. The *Rc* value of the substrate also decreased after the tests by about 45%. Maximum profile peak height – *Rp* after the tests on the coating and the substrate was reduced by 86% and 25%, respectively. The data on the surface profile of the specimens under analysis can be supplemented with the skewness parameter Rsk values. The *Rsk* parameter describes the shape of distribution, that is, whether the data are symmetrically distributed on both sides of the mean plane. The value of this parameter in all cases analysed here was negative, which confirms the left-side asymmetry of the distributions. The values of all the parameters taken into account decreased after the tribological tests.

CONCLUSIONS

The SEM/EDS results analysis showed the consistence of the coating structure with that assumed during the manufacturing process. Surface morphology analysis, point analysis of chemical composition, and the linear distribution of elements analysis all demonstrated that the a-C:H:Si coating is composed mainly of carbon, silicon, and hydrogen, which does not provide any signal in the spectrum analysed. The interlayers of the coating, consisting of silicon and tungsten, improve the adhesion of the coating to the substrate of steel.

The contact angle measurements showed the lowest values for the 100Cr6 disc without the coating and with the a-C:H:Si coating for the ionic liquid to be 67° and 65°, respectively. This indicates that, of all the liquids analysed, the ionic liquid is the best lubricant for the surfaces with a-C:H:Si coating.

After tribological tests with lubrication using trihexyltetradecylphosphonium bis(trifluoromethylsulfonyl) amide, the lowest average coefficient of friction was obtained from the disc coated with a-C:H:Si, $\mu \cong 0.06$. Under dry friction, the a-C:H:Si coating had a friction coefficient more than ten times lower than that obtained from the 100Cr6 steel surface. This results from the self-lubricating properties – the presence of graphite in the DLC coating.

Observation of the wear scars on the discs after tribological tests demonstrated lower wear in the specimens with a-C:H:Si. Analysis of the isometric images and primary profiles of the discs after the tests indicated no wear of the a-C:H:Si specimen. Deeper indentations, about 1.5 µm, were recorded for the 100Cr6 steel disc.

The silicon-doped DLC coating under lubrication with ionic liquid reduces both the friction coefficient and wear of the friction pair under test. Attention should be drawn to the ecological character of the ionic liquid, as shown by its structure and properties, and to the possibility of replacing classical lubricants with trihexyltetradecylphosphonium Bis(trifluoromethylsulfonyl) amide.

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