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EFFECT OF ZDDP AND FULLERENES ADDED TO PAO 8 LUBRICANT ON TRIBOLOGICAL PROPERTIES OF THE SURFACE LAYER OF STEEL BARE STEEL AND W-DLC COATING

WPŁYW DODATKÓW ZDDP I FULERENÓW W OLEJU PAO 8 NA WŁAŚCIWOŚCI TRIBOLOGICZNE WARSTWY WIERZCHNIEJ STALI ORAZ POWŁOKI W-DLC

Key words: fullerenes, PAO 8, wear, W-DLC coating, ZDDP.

Abstract: The paper presents the effect of adding zinc dialkyldithiophosphate (ZDDP) and/or fullerenes on the properties of tribological systems coated with diamond-like carbon coatings doped with tungsten (W-DLC) and 100Cr6 steel without coating. The tribological tests were performed using a ball-on-disc tribometer. Discs made of bare 100Cr6 steel and discs with W-DLC coating were used as samples. Balls made of 100Cr6 steel were used as counter samples. The lubricants used:poly (α) olefin oil PAO 8, PAO 8 + 1.5% of ZDDP, PAO 8+ 0.005% of C60 fullerenes and PAO 8 + 1.5% of ZDDP + 0.005% of C60 fullerenes. Observations of the W-DLC coating were carried out using a scanning microscope, and the EDS analysis enabled the identification of the chemical composition along the cross-sections of the coating. A confocal microscope operating in the interferometric mode was used to analyse the geometrical structure of the samples before and after the friction tests. Observations of the wear marks on the samples and counter-samples were also carried out using a scanning microscope, and the chemical composition on the surfaces of the friction pairs was determined. The obtained test results indicated that the PAO 8 + ZDDP + C60 lubricant caused a reduction in the coefficient of friction and the linear wear in the tested steel friction pairs. In the case of W-DLC coating, however, the ZDDP added to PAO 8 increased the resistance to motion with a simultaneous improvement in anti-wear properties.

Słowa kluczowe: fulereny, ZDDP, powłoka W-DLC, PAO 8, zużycie.

Streszczenie: W artykule zbadano wpływ dodatku w postaci dialkiloditiofosforanu cynku ZDDP i/lub fulerenów na właściwości systemów tribologicznych pokrytych diamentopodobnymi powłokami domieszkowanymi wolframem W-DLC oraz stali 100Cr6 bez pokrycia. Testy tribologiczne wykonano na testerze tribologicznym pracującym w skojarzeniu trącym kula-tarcza. Do badań jako próbek użyto tarcz ze stali 100Cr6 bez powłoki i z naniesioną powłoką W-DLC, a jako przeciwpróbek użyto kul ze stali 100Cr6. Badania przeprowadzono w warunkach smarowania olejem poli(α)olefinowym PAO 8, PAO 8 i 1,5% ZDDP, PAO 8 i 0,005% fulerenów C60 oraz PAO 8 i 1,5% ZDDP oraz 0,005% fulerenów C60. Za pomocą mikroskopu skaningowego wykonano obserwacje powłoki W-DLC, a analiza EDS umożliwiła identyfikację składu chemicznego na przekrojach poprzecznych powłoki. Mikroskop konfokalny z trybem interferometrycznym posłużył do analizy struktury geometrycznej próbek przed i po testach tarciowych. Przeprowadzono także obserwacje śladów wytarcia próbek i przeciwpróbek na mikroskopie skaningowym oraz określono skład chemiczny na powierzchniach par trących. Uzyskane wyniki badań wskazały, że substancja smarowa PAO 8 + ZDDP + C60 spowodowała zmniejszenie współczynnika tarcia i zużycia liniowego w badanych stalowych węzłach tarcia. Natomiast oddziaływanie ZDDP w przypadku powłoki W-DLC i PAO 8 wpłynęło na zwiększenie oporów ruchu z jednoczesną poprawą właściwości przeciwzużyciowych.

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INTRODUCTION

Modern state-of-the-art mechanical systems operate in demanding conditions such as variable loads, speeds, temperatures, and aggressive environments. The frictions pairs, which constitute by far the largest part of all mechanical systems, the mixed lubrication phenomenon often takes place (permanently or temporarily), where there is a direct contact between rubbing surfaces (dry friction) and the surfaces are separated locally by an oil film (boundary friction). Therefore, it is a good solution to apply anti-wear coatings **[L. 1]** and coatings with a low coefficient of friction **[L. 1, 2]**. All the above advantages can be achieved by using DLC coatings, which have become one of the most valuable and promising low friction protective coatings in the last decade **[L. 1]**.

In addition, in recent decades, various synthetic lubricants with optimal rheological and tribological properties for a variety of applications have been developed **[L. 3]**. Polyalphaolefin oil (PAO) is a high-performance synthetic base oil that has been used for many years **[L. 4]**. PAOs are synthetic hydrocarbons with relatively short chains **[L. 3, 5]**.

Compared to conventional mineral oils, PAO has very good viscosity and temperature properties, high temperature of thermal decomposition, high flash point and low volatility. Therefore, PAO has long been one of the most widely used base oils. Properties of PAO can be further improved by introducing additives, especially to reduce friction and wear. One of the most important anti-wear additives is zinc dialkyldithiophosphate (ZDDP). ZDDPs have been widely used since the 1930s and are still added to almost all engine oils as they provide a very good performance under various operating conditions **[L. 4]**.

In the tests described in this paper, fullerenes were added to the tested lubricant to investigate its interaction with ZDDP. Fullerene C60 has been used as an additive to lubricating oil, ionic liquids **[L. 10, 11]**, solid lubricants **[L. 8]** and other solutions **[L. 12–14]** (e.g., ethanol, water, etc.). Many research teams have investigated the tribological properties of fullerene particles as an additive to liquid lubricants. The results showed that the presence of fullerene-styrene copolymer in a water-based liquid enabled the reduction of the coefficient of friction and wear **[L. 6]**.

Liu and co-authors **[L. 7]** investigated the effect of C60 that they added to paraffin oil in steel/steel tribological tests. The tests were performed at various temperatures. Their results showed that the addition of alkylated C60 reduces the coefficient of friction at both 25°C and 90°C. This can be attributed to the presence of aliphatic chains that lead to an improvement in the solubility and dispersibility of the target compound in lubricating oils.

Ku and his team **[L. 8]** investigated the tribological properties of mineral oil as a function of viscosity with the addition of fullerenes. The anti-seize properties were assessed using a four-ball tester. The lubrication tests were carried out with a disc-disc tribometer using various loads. The pitting points (burn, seizure) for all oils with the C60 additive were higher than for pure oil. Also, the diameters of wear marks on the samples lubricated with clean oil and oil + C60 decreased with increasing oil viscosity, and the wear marks on the samples lubricated with all lubricants + C60 were smaller than for pure oil. When using oil with C60 fullerenes, a much lower value of the friction coefficient was obtained than in the case of pure oil, when the oil's viscosity was low and the standard load was high. The authors proved that the addition of fullerenes to the lubricant was more effective when the base oil viscosity was low under higher loading conditions.

The authors of **[L. 9]** analysed the behaviour of lubricants containing zinc dialkyldithiophosphate (ZDDP) and ordered molecular structures, such as carbon nanotubes (CNT) fullerenes (C60) and antistatic additive (ASA). Two types of tribological tests were performed: high-frequency reciprocating rig test (HFRR) and triboelectric test (TET). It was found that the effectiveness of the lubricant additive (ZDDP) depends on the presence of ordered molecular structures that are able to conduct energy from the surface of the sample to the ZDDP reaction zones inside the boundary lubricant layer. It was proved that the CNT and ASA can effectively conduct energy. Fullerenes (C60), however, are not suitable as energy conductors in the process of lubrication.

The research described in this paper aimed to investigate the effect of C60 fullerenes on the tribological properties of the PAO 8 lubricant containing the ZDDP additive and whether C60 has a catalysing effect enhancing the properties of the ZDDP additive. The additional aim of the research was to verify if the lubricants used for the tests affect the chemical composition of the bare steel surface layer and the W-DLC coatings.

RESEARCH MATERIALS AND METHODOLOGY

Steel discs with and without W-DLC coating were used for the tests. The balls used for the tests were made of 100Cr6 steel with a diameter of 6 mm. 100Cr6 is a high carbon steel used for rolling elements of bearings, such as balls, rollers and races. This grade of steel has very good resistance to wear and fatigue and the stability of elasticity and microstructure at extreme temperatures **[L. 10]**. **Table 1** shows the chemical composition of 100Cr6 steel.

W-DLC coatings have very good tribological properties **[L. 11, 12]**, good corrosion resistance and high hardness. The coatings have properties very similar to the properties of diamonds **[L. 13]**. Thanks to these advantages, W-DLC coatings are used in many industrial applications, e.g. for the production of cutting tools **[L. 11, 12]**.

Table 1. Composition of 100Cr6 steel
Tabela 1. Skład chemiczny stali 100Cr6

Element	%
C	0.93 – 1.05
Mn	0.25 – 0.45
Si	0.15 – 0.35
P	< 0.025
S	< 0.030
Cr	1.35 – 1.6
Cu	< 0.30

The polyalphaolefin PAO 8 was used as a lubricant for the tests. PAO 8 is a synthetic oil with a hydrocarbon structure (isoparaffins), produced by catalytic oligomerisation of linear α-olefins. The basic properties of PAO8 oil are given in **Tab. 2**. 1.5% of ZDDP additive was introduced to PAO 8 oil, the basic properties of which are given in **Tab. 3**. The following marking of the tribological tests lubricants are used further in this paper:

- PAO 8 – synthetic polyalphaolefin oil,
- ZDDP – anti-wear additive of zinc dialkyldithiophosphate, added in an amount of 1.5% (by weight)
- C60 – fullerenes that were added in an amount of 0.005% (by weight).

The tribological tests were performed using a ball-on-disc TRB tribometer[3]. The parameters of the tests performed are summarised below:

- load P = 10 N,
- slip speed v = 0.1 m/s,
- sliding distance s = 1000 m,
- humidity 36 ± 10%,
- ambient temperature $T_0 = 24 \pm 4°C$,
- friction pair: 100Cr6 steel ball – 100Cr6 steel disc, 100Cr6 steel ball – 100Cr6 steel disc with W-DLC coating.
- lubricant: PAO 8, PAO 8 + ZDDP, PAO 8 + C60, PAO 8 + ZDDP + C60.

Table 2. Properties of PAO 8
Tabela 2. Właściwości oleju PAO 8

Properties	Value
Specific gravity at 15.6°C	833 kg/m^3
Kinematic viscosity at 100°C 40°C -40°C	 7.8 mm^2/s 46.4 mm^2/s 19 570 mm^2/s
Viscosity Index	138

Table 3. Properties of the commercial zinc-dithiophosphate (ZDDP) lubricant additive
Tabela 3. Podstawowe właściwości ZDDP

Properties	**value**
Density (25^{o}C)	1160 kg/m^3
Kinematic viscosity at 40^{o}C	150 mm^2/s
Zn content	9,0% weight
P content	8,5% weight
S content	16,5% weight

In the tribological characteristics, linear wear, friction coefficient, and wear trace area were assessed. With the help of a Leica DCM8 confocal microscope operating in interferometric mode, the signs of wear on the discs and balls were observed. A scanning electron microscope Phenom XL equipped with an EDS microanalyser was used for checking the chemical composition of samples and counter-samples before and after tribological tests. Additionally, the chemical composition of the W-DLC coating was examined using the microscope.

RESEARCH RESULTS AND DISCUSSION

The chemical composition of the W-DLC coating is shown in **Figure 1**. The analysis of the chemical composition of the disc with the W-DLC coating showed that the coating consists of carbon and tungsten and that the interlayer is made of chromium (**Figure 1**). The purpose of the interlayer is to increase the adhesion of the coating to the substrate.

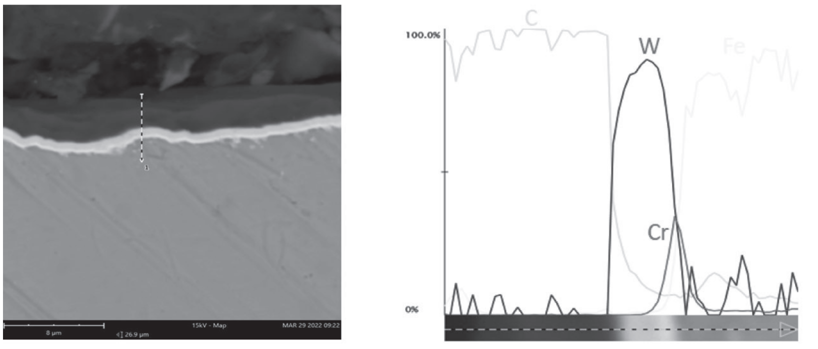


Fig. 1. Cross-sectional view of the disc structure with EDS patterns
Rys. 1. Analiza liniowa powłoki W-DLC

Figure 2 summarises the tribological test curves in the form of diagrams of friction coefficients and linear wear as a function of the friction path for the tested material pairs **[L. 14]**.

Based on the test results, it was noticed that the lowest value of the friction coefficient was recorded for the disk with W-DLC coating in the PAO 8 lubrication test and the highest also for the disc with W-DLC coating in the PAO 8 +C60 lubrication test. The lowest value of linear wear was noticed for the uncoated steel disc with the PAO 8 + ZDDP lubrication and the highest for the W-DLC coated disc during the test with the PAO 8 lubrication.

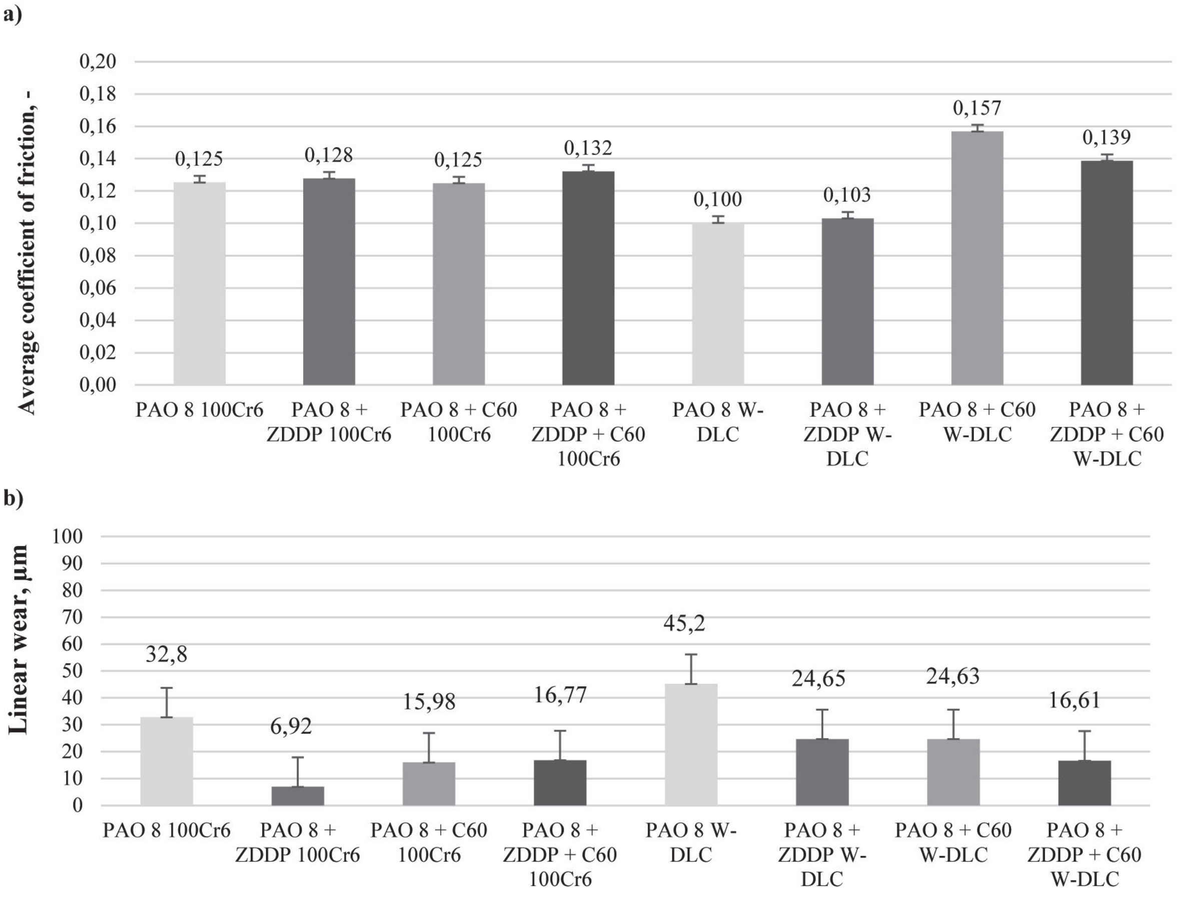


Fig. 2. The results of the tribological tests: a) average coefficient of friction and b) linear wear
Rys. 2. Wyniki badań tribologicznych: a) średni współczynnik tarcia i b) zużycie liniowe

The analysis of the tests carried out using a TRB tribometer indicates that the presence of the W-DLC coating completely changes the chemical mechanism of action of the lubricating additives (ZDDP) against the steel surface. Moreover, ZDDP exhibits limited tribological activity in contact with the W-DLC coating surface and high efficiency in contact with the steel surface (when in contact with the W-DLC surface, C60 catalyses the action of ZDDP). ZDDP has shown a very high anti-wear activity in contact with the steel surface. Moreover, in contact with the steel surface, the activity of ZDDP is inhibited by C60 fullerenes. It was observed that C60 increases the value of the friction coefficient when in contact with steel or W-DLC, but this effect is more significant for the W-DLC surface.

Before and after the tribological tests, the discs and balls were analysed using a confocal microscope operating in the interferometric mode. The results of these tests are shown in **Figure 4** and **Figure 5**. **Figure 3** shows the isometric images and primary profiles obtained for the discs with the W-DLC coating and without the coating and the ball prior to the tribological tests.

Figure 4 shows isometric images and primary profiles obtained for the discs and balls after friction tests with pure PAO 8 lubrication.

Figure 5 shows isometric images and primary profiles obtained for the discs and balls after friction tests with the PAO 8 + ZDDP lubricant.

Figure 6 shows isometric images and primary profiles obtained for the discs and balls after friction tests with the PAO 8 + C60 lubricant.

Figure 7 shows isometric images and primary profiles obtained for the discs and balls after friction tests with the PAO 8 + ZDDP + C60 lubricant.

When analysing the maximum depth and the maximum area of the wear marks on the discs, the largest marks were found on the disc with the

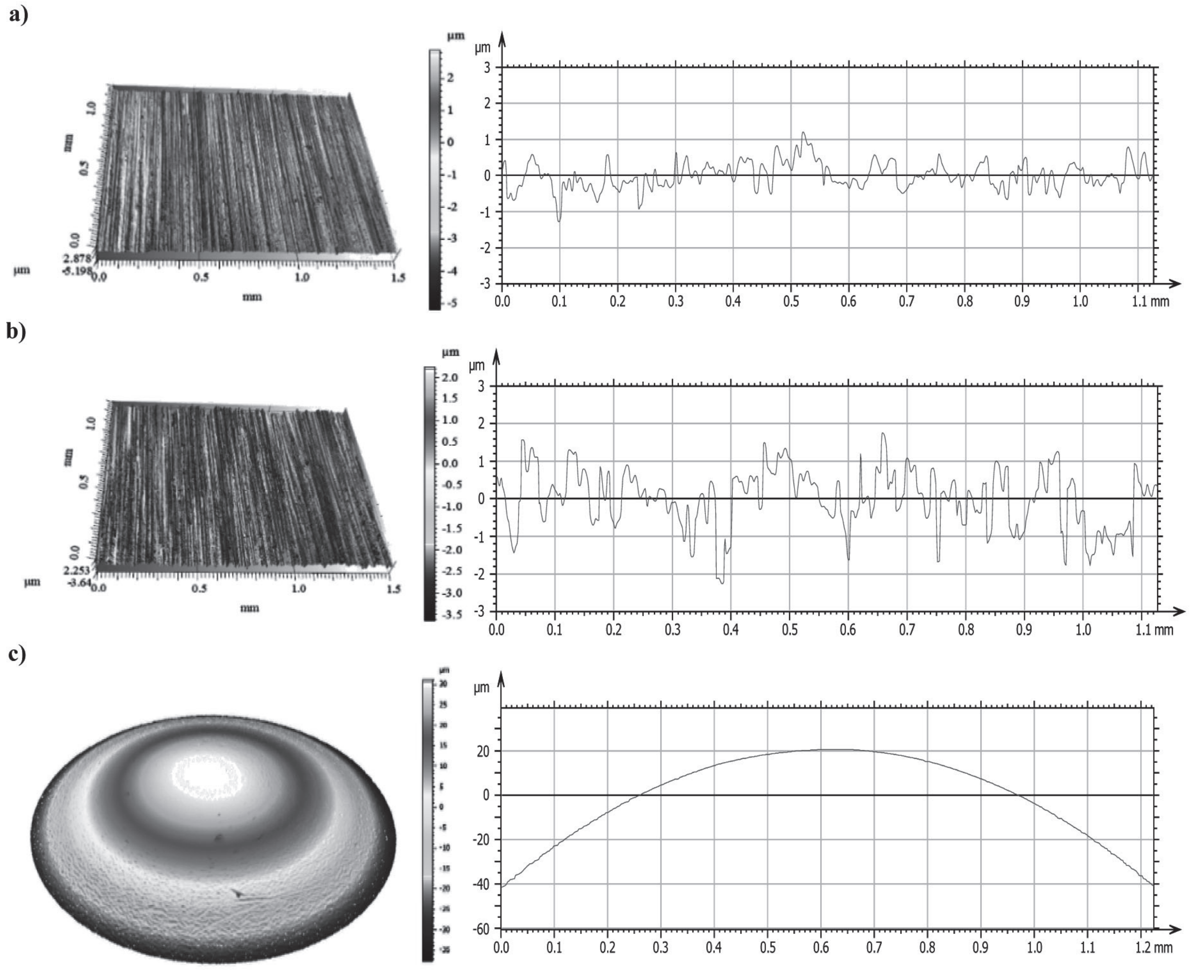
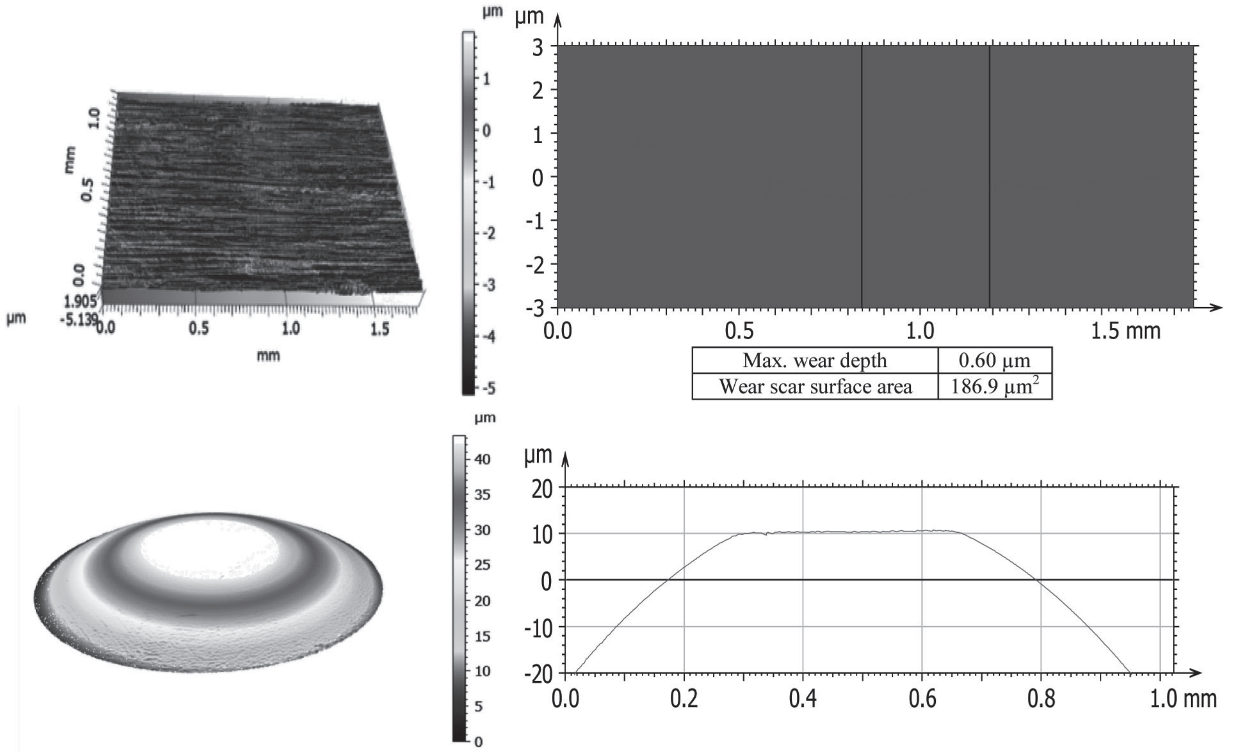


Fig. 3. Surface textures before the tribological tests: isometric views and primary profiles of: a) the W-DLC coated steel disc, b) uncoated steel disc, c) 100Cr6 steel ball

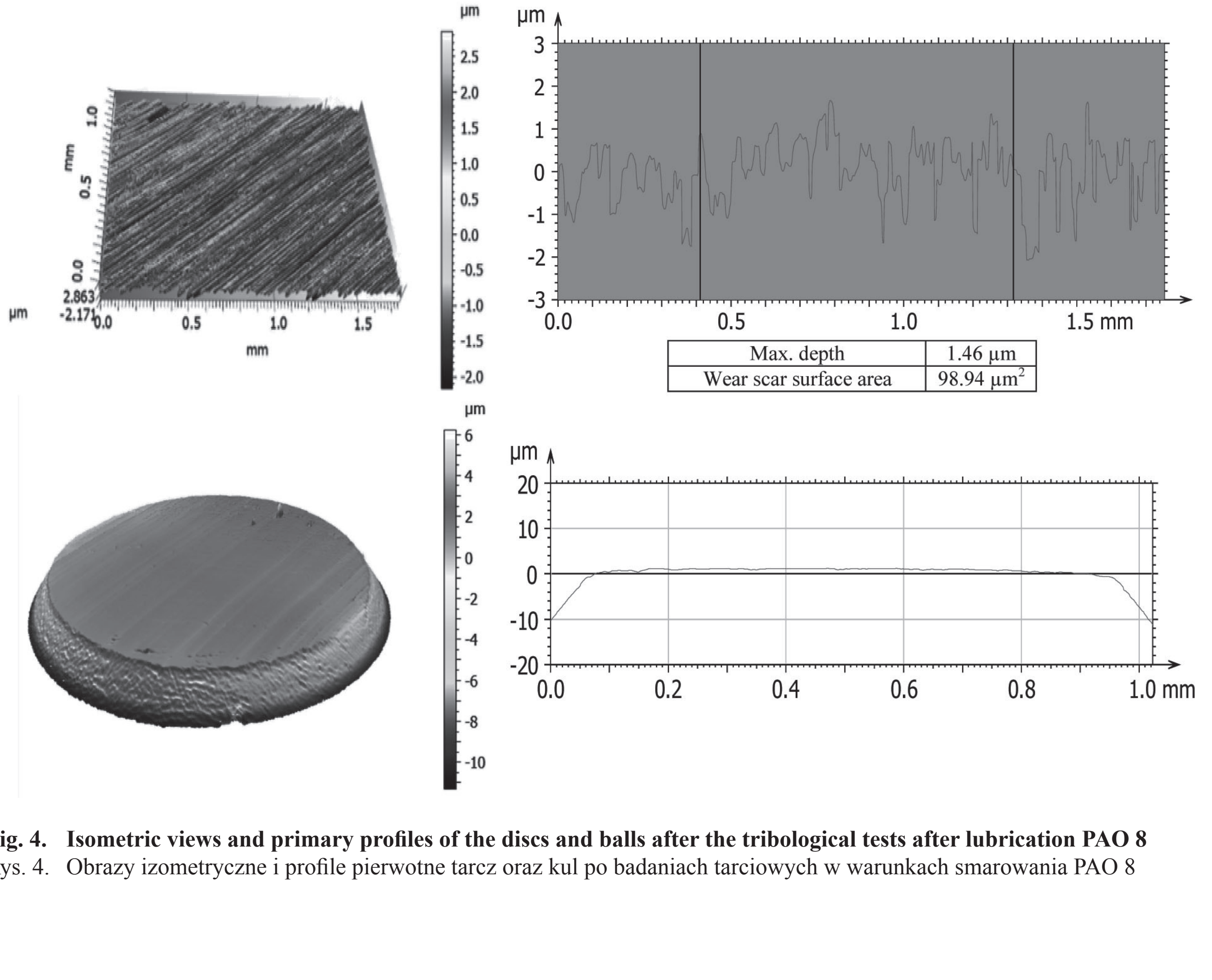
Rys. 3. Struktura geometryczna powierzchni przed badaniami tribologicznymi: obrazy izometryczne i profile pierwotne: a) tarczy stalowej z powłoką W-DLC, b) tarczy stalowej, c) kuli ze stali 100Cr6

a) PAO 8 **[L. 12]**



Max. wear depth	0.60 µm
Wear scar surface area	186.9 µm^2

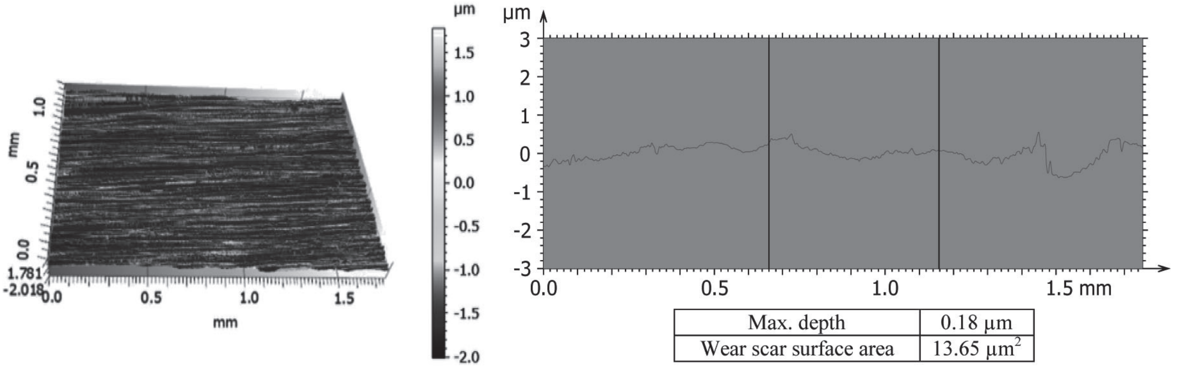
b) PAO 8 **W-DLC [L. 12]**



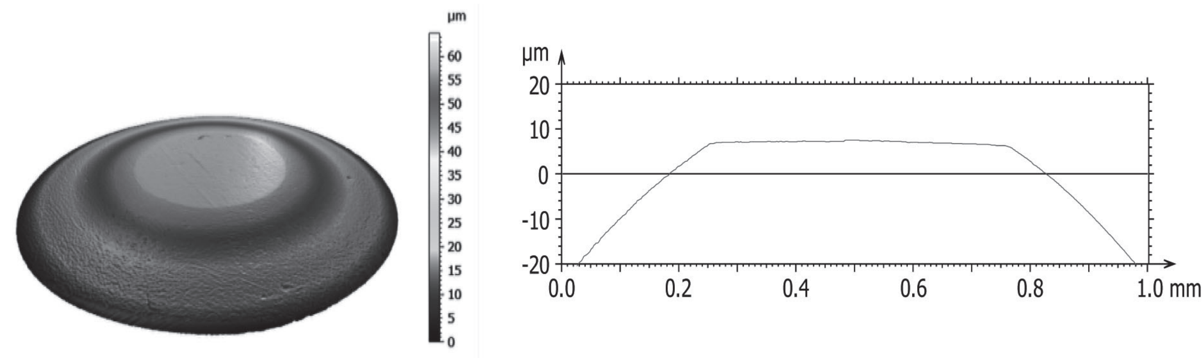
Max. depth	1.46 µm
Wear scar surface area	98.94 µm^2

Fig. 4. Isometric views and primary profiles of the discs and balls after the tribological tests after lubrication PAO 8
Rys. 4. Obrazy izometryczne i profile pierwotne tarcz oraz kul po badaniach tarciowych w warunkach smarowania PAO 8

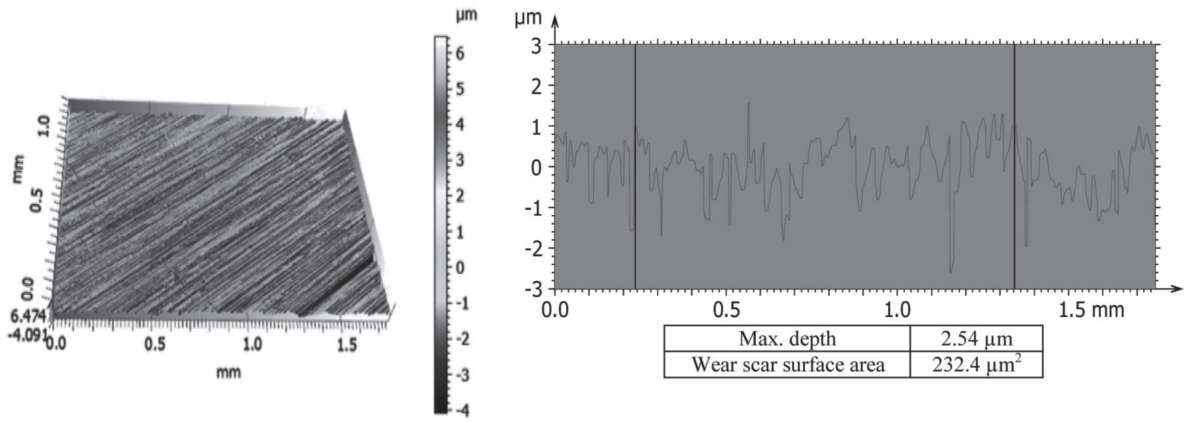
a) PAO 8 + ZDDP **[L. 12]**



Max. depth	0.18 µm
Wear scar surface area	13.65 µm²



b) PAO 8 + ZDDP **W-DLC [L. 12]**



Max. depth	2.54 µm
Wear scar surface area	232.4 µm²

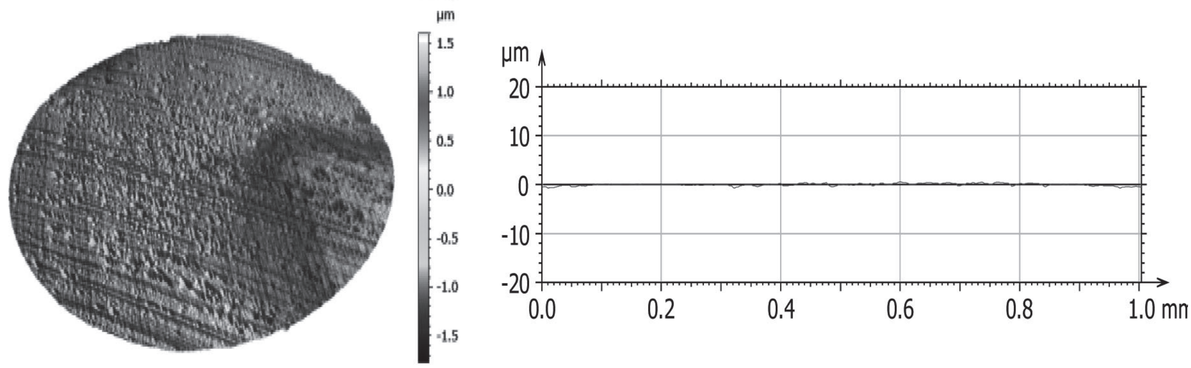
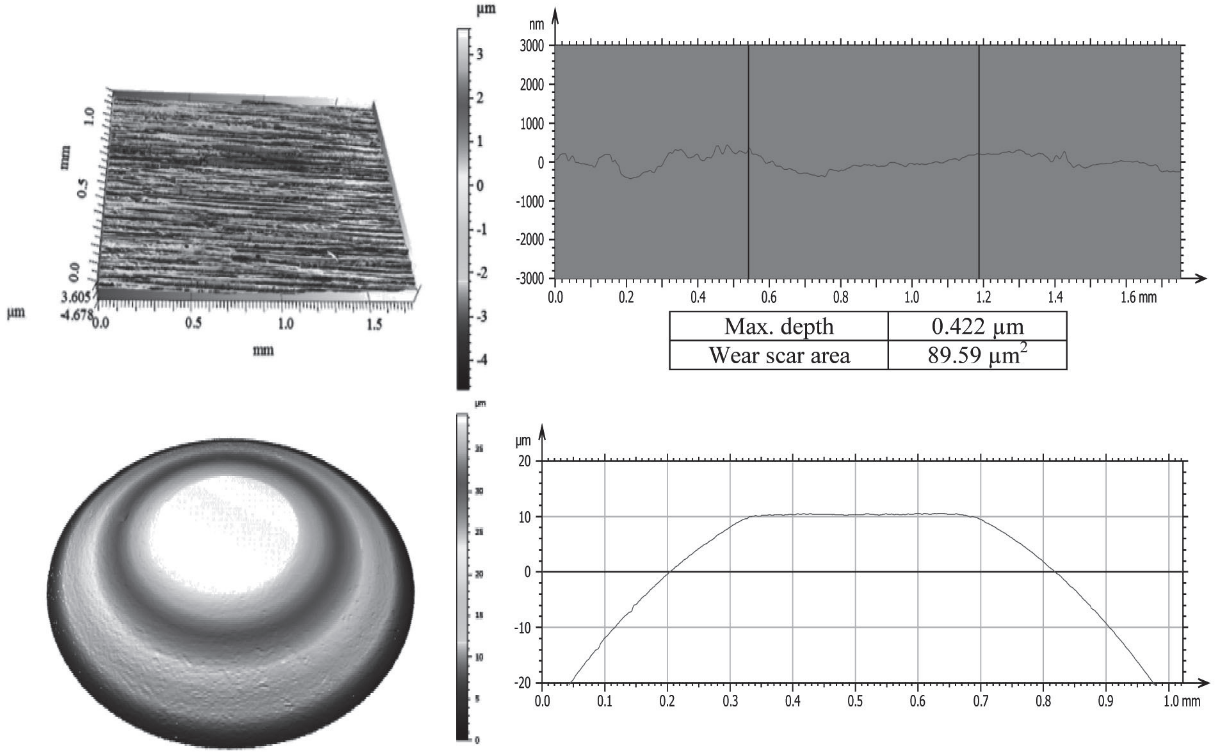


Fig. 5. Isometric views and primary profiles of the discs and balls after the tribological tests after lubrication PAO 8+ ZDDP

Rys. 5. Obrazy izometryczne i profile pierwotne tarcz oraz kul po badaniach tarciowych w warunkach smarowania PAO 8 + ZDDP

a) PAO 8 + 0.005% C60



b) PAO 8 + 0.005% C60 **W-DLC**

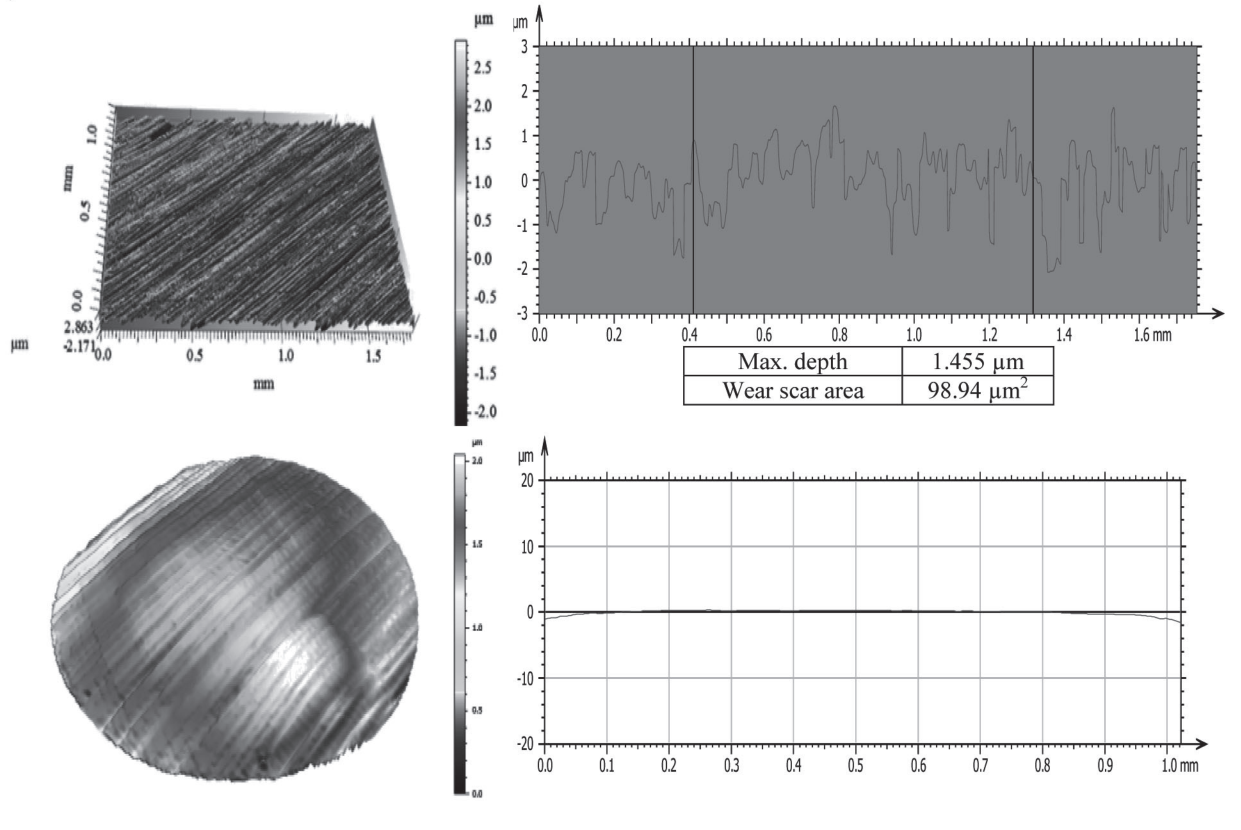


Fig. 6. Isometric views and primary profiles of the discs and balls after the tribological tests after lubrication PAO 8 + C60

Rys. 6. Obrazy izometryczne i profile pierwotne tarcz oraz kul po badaniach tarciowych w warunkach smarowania PAO 8 + C60

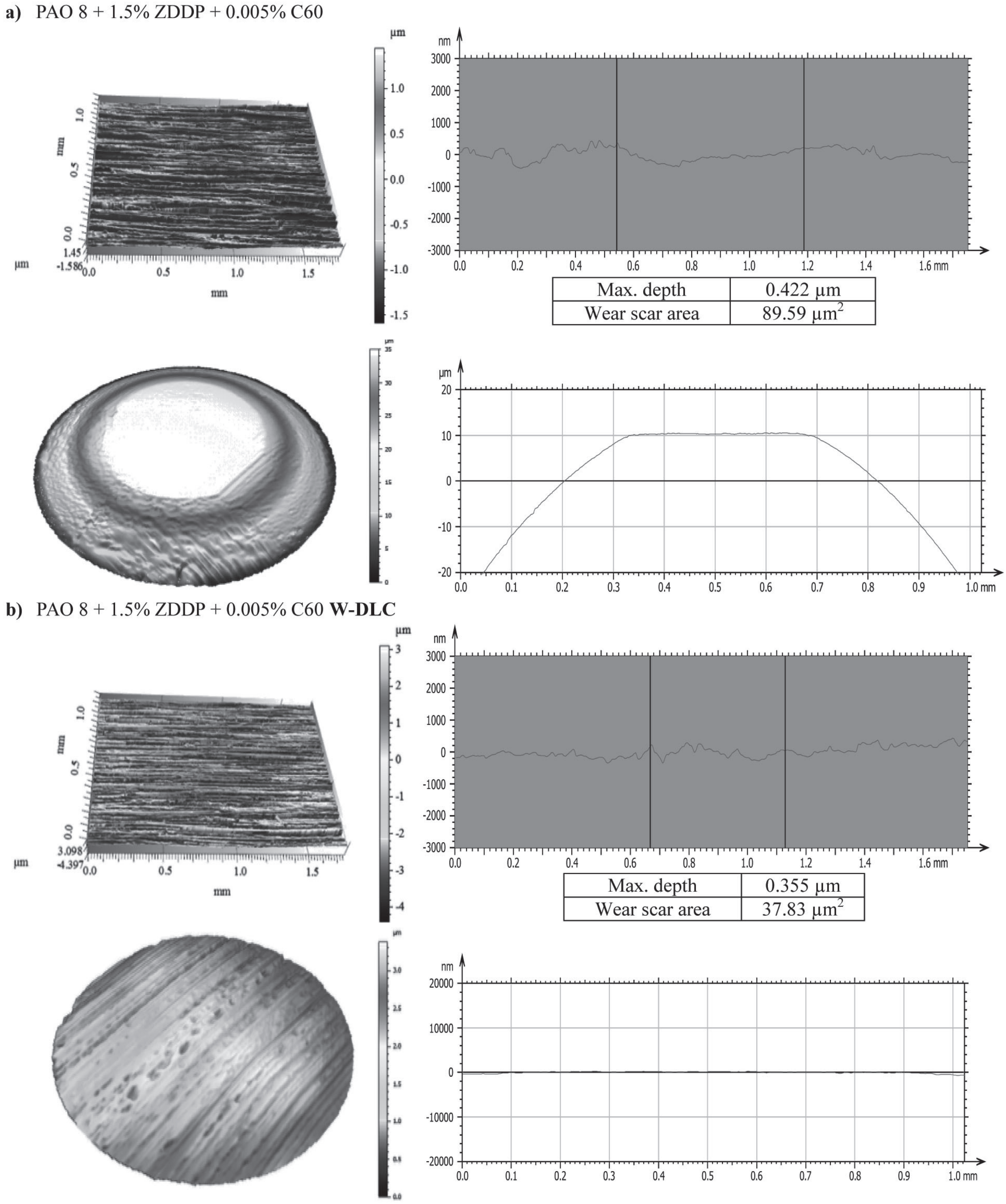


Fig. 7. Isometric views and primary profiles of the discs and balls after the tribological tests after lubrication PAO 8 + ZDDP + C60

Rys. 7. Obrazy izometryczne i profile pierwotne tarcz oraz kul po badaniach tarciowych w warunkach smarowania PAO 8 + ZDDP + C60

W-DLC coating lubricated during the test with the PAO8 + ZDDP lubricant. The wear marks observed were 2.54 μm and 232.4 μm^2, respectively. The smallest maximum wear depth (0.18 μm) and wear area (13.65 μm^2) were obtained for the bare steel disc lubricated with the PAO8 + ZDDP test lubricant. What is more, the wear mark on this disc was the narrowest in this case.

Tables 4 and **5** summarise the roughness of the discs and balls before and after tribological tests.

Table 4. Surface texture parameters for the uncoated steel discs and steel balls before and after the tribological tests for PAO 8 + ZDDP

Tabela 4. Parametry chropowatości powierzchni dla tarcz stalowych bez powłoki i kul przed oraz po testach tribologicznych dla PAO 8 +ZDDP

Surface texture parameters	Before test		After test					
			PAO 8+ ZDDP		PAO 8+C60		PAO 8 + ZDDP + C60	
	disc	ball	disc	ball	disc	ball	disc	ball
Sa [µm]	0.39	0.15	0.37	2.09	0.31	10.39	0.30	8.64
Sq [µm]	0.50	0.22	0.46	2.46	0.39	11.88	0.37	10.05
Sp [µm]	1.45	1.51	1.19	4.89	1.45	16.14	1.02	17.36
Sv [µm]	1.79	4.25	1.44	7.71	1.59	23.17	1.16	19.03
Sz [µm]	3.23	5.76	2.63	12.61	3.04	39.32	2.18	36.39
Ssk [-]	-0.62	-1.43	-0.26	-0.14	-0.13	-0.13	-0.22	-0.01
Sku [-]	3.55	18.59	2.68	2.05	2.81	1.72	2.63	1.82

Table 5. Surface texture parameters for the steel disc with coating W-DLC and steel balls before and after the tribological tests for PAO 8 + ZDDP

Tabela 5. Parametry chropowatości powierzchni dla tarcz stalowych z powłoką W-DLC i kul przed oraz po testach tribologicznych dla PAO 8 +ZDDP

Surface texture parameters	Before test		After test					
			PAO 8 + ZDDP		PAO 8+C60		PAO 8 + ZDDP + C60	
	disc	ball	disc	ball	disc	ball	disc	ball
Sa [µm]	0.57	0.15	0.40	6.24	0.54	0.17	0.45	0.13
Sq [µm]	0.71	0.22	0.53	7.47	0.73	0.19	0.58	0.15
Sp [µm]	2.19	1.51	1.74	16.78	3.50	0.33	1.94	0.25
Sv [µm]	3.53	4.25	5.01	20.71	2.25	0.24	3.88	0.32
Sz [µm]	5.72	5.76	6.76	37.49	5.75	0.58	5.82	0.55
Ssk [-]	-0.19	-1.43	-0.40	-0.11	-0.31	0.10	0.04	-0.14
Sku [-]	2.91	18.59	5.94	2.33	3.77	1.65	3.49	1.95

When comparing the bare steel discs' geometrical surface parameters (**Table 4**), it was noticed that the values of Sq, Sp, Sv, Sz and Sku were lower when the PAO 8 + ZDDP + C60 lubricant was used, which indicated that the disc surface was smoothed during the tribological test. When comparing the geometrical parameters of the W-DLC coated discs surface (**Table 5**), it was noticed that the values of Sa, Sq, Sp were lower when the PAO 8 + ZDDP lubricant was used. In turn, the PAO 8 + C60 lubricant provided lower values of such parameters as Sv and Sz. The above is proved by large differences between the peaks and valleys on the tested disc on the worn spot. The Ssk parameter was negative in almost all of the cases considered (except for the W-DLC-coated disc lubricated with the PAO 8 + ZDDP + C60 lubricant, where this value was very close to zero), which is suggested by the flat surface with slightly inclined and rounded peaks **[L. 13]**. The Ssk parameter for all test cases was close to 3, i.e., the normal distribution.

During the friction test using the bare steel disc lubricated with PAO 8 + ZDDP, lower values of the Sa, Sq, Sp, Sv and Sz parameters were obtained for the ball. The surface of the ball was superfinished during the tests. During the friction tests using the W-DLC coated discs lubricated with the PAO 8 + ZDDP + C60 lubricant, the ball's surface was also superfinished, and lower values of the following parameters were recorded: Sa, Sq, Sp, Sz.

Figure 8 summarises the results of the wear mark chemical composition analysis at selected spots of the bare steel disc.

Figure 9 summarises the results of the wear mark chemical composition analysis at selected spots of the W-DLC coated disc.

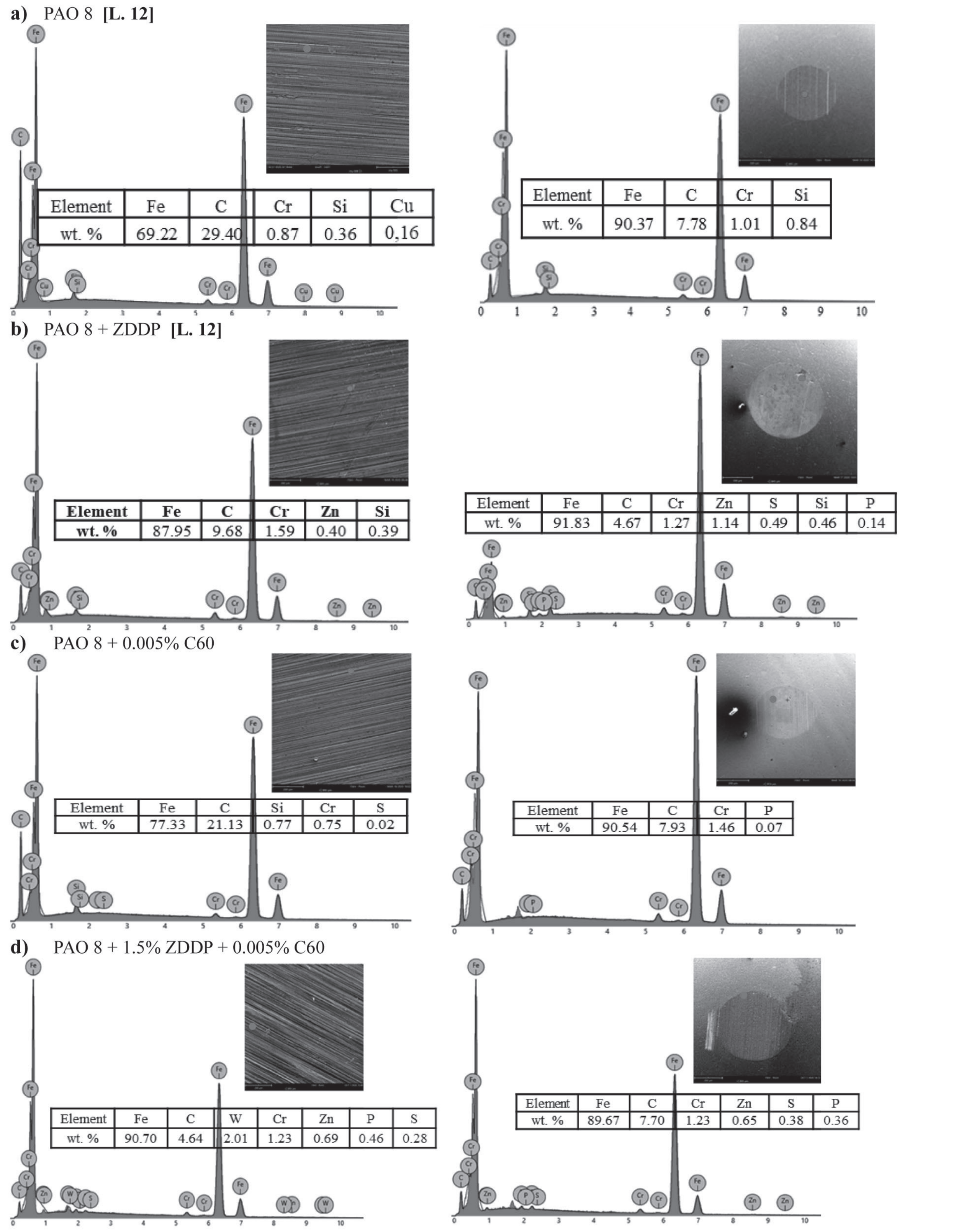


Fig. 8. EDS patterns for different points of the wear track resulting from the sliding contact of the uncoated steel discs with the steel balls

Rys. 8. Widmo charakterystycznego promieniowania rentgenowskiego z obszaru wytarcia po kontakcie ślizgowym tarcz stalowych ze stalowymi kulkami

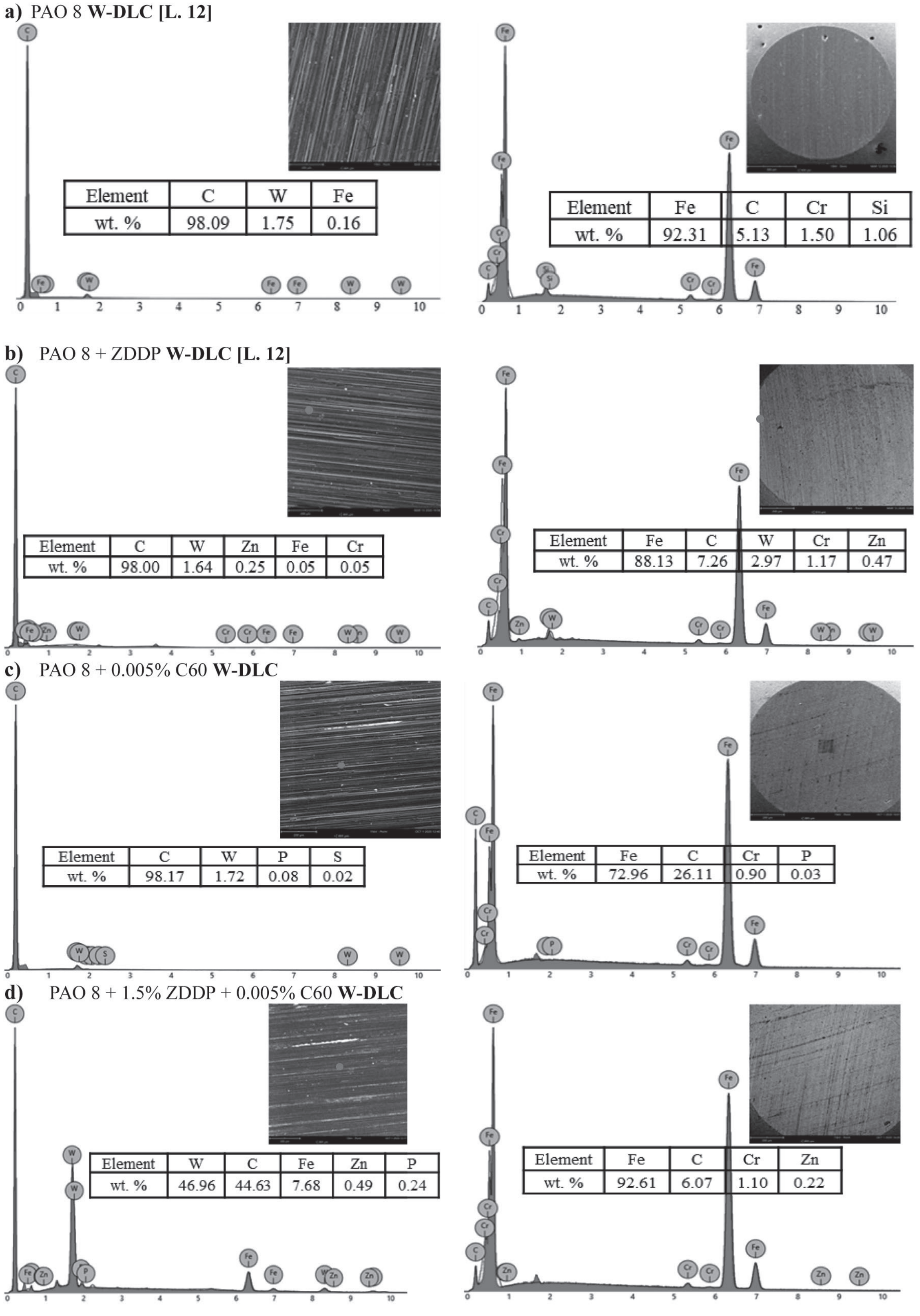


Fig. 9. EDS patterns for different points of the wear track resulting from the sliding contact of the coated steel discs with the steel balls

Rys. 9. Widmo charakterystycznego promieniowania rentgenowskiego z obszaru wytarcia po kontakcie ślizgowym tarcz z powłoką W-DLC ze stalowymi kulkami

The chemical composition was measured by determining the presence of elements such as C, W and Cr that constitute the coating. Fe, C, Cr and Si (a small amount of S and P – close to 0) are elements that originate from 100Cr6 steel, and Zn and a larger amount of P and S originate from the ZDDP additive.

When analysing the chemical composition tests results of the wear marks both on the balls and the discs, it was observed that in the cases of friction tests using the bare steel samples with the PAO 8 + ZDDP + C60 lubricant, more accumulated zinc and sulphur was discovered than on the disc coated with W-DLC. These elements are derived from the lubricant used for the tests. An anti-wear layer has been formed. Comparing the bare steel discs after the friction tests with the PAO 8 + ZDDP lubricant to the test with the PAO 8 + ZDDP + C60 lubricant, it was observed that after the tests with the lubricant containing C60, a higher concentration of zinc, phosphorus and sulphur atoms was found.

The wear on the bare steel disc was much lower (approx. 10.8 µm) than on the W-DLC coated disc (approx. 72 µm).

CONCLUSIONS

The main aim of the research was to evaluate the influence of C60 fullerenes on the tribological properties of a lubricant containing ZDDP enhancing additives introduced to the synthetic base oil.

Based on the research, the following were established:

1. As a result of the tests, it was found that the PAO 8 + ZDDP + C60 lubricant in combination with the W-DLC coated disc resulted in a reduction of linear wear compared to other lubricants (PAO 8, PAO 8 + ZDDP and PAO 8 + C60).
2. Whereas the lowest values of the linear wear during the friction tests with the use of the bare steel discs performed in the same tribological conditions were obtained when the PAO 8 + ZDDP lubricant was used.
3. The use of the ZDDP additive in the tested lubricants resulted in a reduction of the abrasion marks volume, both on the steel discs coated with W-DLC and on the bare steel discs.
4. Based on the analysis of the tribological system elements, it was found that when the PAO 8 + ZDDP + C60 lubricant is used, an anti-wear surface layer is created with increased content of zinc atoms and a smaller content of sulphur and phosphorus atoms.
5. It was found that the PAO 8 + ZDDP + C60 lubricant improved the parameters of the geometrical structure of the surface.

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