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Evaluation of the effects of the application of Ti−C:H DLC coatings obtained by PVD techniques in the kinematic pairs of internal combustion engines and powertrain systems

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The article attempts to analyze the possible effects of using Ti−C:H DLC carbon coatings produced by pulsed magnetron sputtering (PVD) to reduce friction coefficient and wear in kinematic pairs found in internal combustion engines and powertrain systems used in automotive vehicles. The aim of such action is primarily to reduce internal losses in the aforementioned units. The coatings were deposited on heat-treated bearing steel 100Cr6, and examined using a scanning electron microscope FEI Quanta 200 Mark II with the chemical analyzer EDS EDAX Genesis XM 2i, tribotester T−01M examining the friction coefficient in the ball-disc correlation and Hommel Werke T8000 profilometers, additionally, in order to check the coating thickness, studies were carried out using the Calotest method. The results obtained indicate that both the friction coefficient and wear are drastically reduced concerning samples on which no DLC coatings were applied.

Key words: *pulsed magnetron sputtering, diamond-like carbon, friction coefficient, wear rate*

1. Introduction

Air pollution [17], and the associated threats to human life, have become an urgent problem to be solved, as it is a cause of premature deaths in the EU [1, 12, 23]. In addition, road transport is one of the main factors that contribute to emissions of pollutants.

For several decades, steps have been taken to create a more "green" transport, for this purpose a several regulations and legal regulations have been introduced that define the requirements that vehicles must meet [10, 13, 21, 24, 28, 32, 33, 39].

It is expected that new, increasingly stringent emission standards will influence vehicle manufacturers to design and implement increasingly sophisticated and effective exhaust cleaning systems [6]. Taking into account that many issues regarding "zero emission" transport are still unresolved (e.g. related to the availability and distribution of CO_2-free electricity, charging infrastructure for electric vehicles in cities, sourcing of raw materials for battery production), vehicles using combustion engines will continue to be produced and sold in the form of hybrid drive systems, which consume less fuel and therefore have lower emissions of pollutants into the atmosphere [6, 31].

The energy and material losses resulting from friction processes and increased wear are a major economic and environmental burden for the entire world [14]. Considering that passenger cars in the world consume 350 million tons of oil annually to overcome friction, which also has an impact on CO_2 emissions and increased costs resulting from the regeneration and replacement of worn parts, the pursuit of minimizing friction and wear becomes a necessity [14]. Many authors support this opinion, arguing further that the automotive industry is moving not only towards increased efficiency, lower fuel consumption, improved reliability, and more environmentally friendly products, but also towards less lubricants [5, 18, 25].

The automotive industry is one of the most important users of modern surface technologies, which improve the materials and products used, making it possible to introduce new and innovative solutions based on the special properties of thin coatings. The use of appropriate coatings makes it possible to change the surface properties in the context of [3]:
- mechanical (frictional wear, frictional resistance)
- chemical (corrosion)
- electrical (conductivity)
- optical (transmittance, reflection, absorption, aesthetic effect).

Examples of attempts to use coatings for mechanical purposes in the automotive industry are primarily:
- Piston rings – coated with chrome galvanically, chromium nitride, titanium nitride in PVD techniques [3, 18, 36, 38, 40]
- Cylinders in engine blocks (carbon steel reinforced with ceramics – thermal spraying) [3]
- Crankshaft bearings – Al−Sn, Al−Sn−Bi alloys [3]
- Ball pivots – plasma nitriding, oxidation [3, 38]
- Injector needles in gasoline engines – carbon DLC coatings – PVD [3, 35, 38]
- Piston pins – carbon DLC coatings – PVD [3]
- Pushrods – carbon DLC coatings – PVD [3, 18]
- Camshaft journals and camshafts – carbon DLC coatings – PVD [3, 18, 22]
- Valves – carbon DLC coatings – PVD [3, 22]
- Gearboxes – carbon DLC coatings – PVD [3, 18, 19, 22, 38].

Continuous research is being conducted, in which attempts are being made to create thin-film coatings for automotive applications, for example based on metal-doped DLC (diamond-like carbon) coatings, or boron carbide [16, 26, 34, 43, 44]. However, an aspect that is increasingly being paid attention to is the cooperation of the proposed thin-film coatings with currently used lubricants and their additives in order to minimize friction [5, 16, 19, 25, 27, 34, 40, 41]. Lubricants have been developed and optimized for the cooperation of metallic surfaces in kinematic pairs used in the automotive industry, while solutions considering "improved surfaces" through applied thin-film coatings are not yet commercialized [25, 34].

Engineers often treat thin-film coating technology as a backup tool, not optimized for design. It happens that this solution is applied to unsuitable substrates (e.g. too low substrate hardness), which results in lesser use of the potential of the applied coating [18]. This technology should be already included in the design phase, so that it becomes a design tool, and it is possible to optimize its use, to achieve maximum benefits [18].

Over the past few decades, many coatings have been developed for thin-film technologies, used both to reduce frictional wear, and losses associated with friction, or to improve corrosion resistance. Metal coatings are used (e.g. based on elements such as Cr, Ni, and Ti), as well as those based on metal nitrides (e.g. CrN, TiN, and TiAlN AlN), metal carbides (e.g. TiC, WC), as well as metal carbonitrides (e.g. TiCN, TiAlCN). Another group that has found application is diamond-like carbon coatings (DLC), which are characterized by high wear resistance and hardness and a low friction coefficient [29].

DLC coating is a type of amorphous carbon coating, whose properties are determined by the content of sp^3 bonds (similar to diamonds) and sp^2 bonds (similar to graphite) and the content of hydrogen [3, 25, 29]. DLC coatings can be divided into those without hydrogen (ta−C, a−C), coatings with hydrogen (ta−C:H, a−C:H) and doped with both metals (a−C:H:M), and non-metallic elements (a−C:H:X) [3, 25, 29].

In automotive engineering, due to their properties, a−C:H and a−C:H:M coatings are most commonly used [25]. a−C:H coatings, despite their high hardness and wear resistance, are characterized by high internal stresses, which can cause problems with obtaining adequate adhesion to the substrate [4]. In the case of metal-doped coatings (a−C:H:M), internal stresses are reduced due to the decrease in coating hardness, thus reducing the problem with adhesion to the substrate. They still show a small friction coefficient, but their wear resistance decreases [4]. The disadvantages of DLC coatings include changing wear and tribological properties with increasing temperature. From a temperature of 100°C, these properties begin to deteriorate, and in the range of high temperatures, hydrogen begins to be released from the DLC matrix [15, 25, 37]. Therefore, the use of such coatings in conditions where high operating temperature dominates is problematic [25]. It should also be mentioned that the technology for producing DLC coatings is expensive, and it is crucial to develop a method in which high repeatability, efficiency and reliability are achieved at the lowest possible cost [20].

Taking into account the excellent tribological and wear resistant properties of DLC coatings and the possibility of modifying these properties by changing the parameters of the layer deposition process and using additives in the form of metals and non-metals, the adaptability of such a solution is high, however, it is necessary to optimize the coating for specific applications.

2. Experimental details

To analyze the possibilities and effects of using DLC coatings in kinematic pairs used in internal combustion engines and vehicle systems, an attempt was made to investigate how these coatings would affect the change in tribological properties in kinematic pairs made of bearing steel 100Cr6 after heat treatment under dry friction conditions.

For research purposes, a a−C:H:M coating was planned, in which the metal used to modify the DLC coating was titanium (Ti−C:H). These studies aimed to determine the parametric properties of the applied coatings and to verify how they would affect the change in the friction coefficient and wear in a kinematic pair made of 100Cr6 steel.

It was assumed that the Ti−C:H coating would be applied to both elements (sample and counter-sample) and only to the sample, while the counter-sample would remain unchanged. The deposition temperature was limited to 200°C in order not to cause structural changes in the base material and a target coating thickness of about 1µm was planned. Lower deposition temperature of the coating may affect its adhesion to the substrate and deteriorate its properties. Therefore, the study aims to investigate the properties of the coating obtained at a lower deposition temperature.

2.1. The technology of applying Ti-C:H coatings

The substrates on which the coatings were applied were made of 100Cr6 steel after heat treatment (hardening and tempering). They were cylindrical plates with a diameter of 28 mm and balls with a diameter of 10 mm. The samples were polished to achieve a roughness of Ra < 0.02 mm. The substrate preparation process also required washing in an alkaline bath and deionized water using ultrasonic cleaners.

Table 1. Composition of 100Cr6 (wt.%) [2]

Fe	C	Cr	Mn	Si	Cu	Ni	Mo	Al
Bal.	0.97	1.38	0.28	0.28	0.21	0.18	0.06	0.04

Prepared samples were placed in a vacuum chamber on a rotating table, in planetary rotation holders at a distance of 10 cm from the source. The coatings were applied by reactive magnetron sputtering using a titanium target. The chamber was initially pumped down to a pressure of 2×10^{-3} Pa to remove oxygen, then the pressure was increased by controlled introduction of argon, until a working pressure of 0.3 Pa was reached.

The first step in the coating deposition process is ion cleaning, which is used to remove oxides and prepare the substrates for coating. The process parameters are: working pressure of 0.5 Pa, substrate polarization voltage of −600 V, arc discharge current of 85 A, and duration of 8 minutes. During the deposition of the target coating, the substrate polarization voltage was maintained at −90 V, and the sputtering power on the titanium target was 1500 W. The process temperature was 150°C, acetylene flow was 18 SCCM, and the duration was 75 minutes.

2.2. Characterization methods

The thickness of the coatings was determined using the Calotest method, i.e. the spherical grinding method according to DIN EN 1071-2:2003.

The adhesion of the obtained coatings was checked using the scratch method on a Revetest Scratch Tester device.

The surface roughness of the obtained coatings was investigated using a Hommel Werke T8000 profilometer, repeating the measurement five times for each sample.

The surface morphology and composition analysis were performed on a FEI Quanta 200 Mark II scanning electron microscope with an EDAX Genesis XM 2i chemical analyzer.

The verification of the microhardness of the obtained coatings was checked using a Fischerscope HM 2000 microhardness tester according to ISO 14577.

The tribological tests were carried out on a T-01M device in a ball-disc combination according to ASTM G 99 and DIN 50324. The following parameters were adopted:

– Load: 20 N
– Speed: 0.2 m/s
– Distance: 1000 m
– Wear radius: 0.01–0.013 m
– Number of revolutions: 12243–15915
– Test duration: 5000 s
– Ball diameter: 0.01 m.

2.3. Results and discussion

Morphological studies of the surface conducted on a scanning electron microscope showed that no defects such as cracks, local delaminations, or chips were present in the applied coatings (Fig. 1). The presence of microdroplets was also observed on the surface of the applied coatings, which can lead to a deterioration of tribological properties [8, 14]. The size of these microdroplets is variable, with diameters of up to several micrometers being recorded.

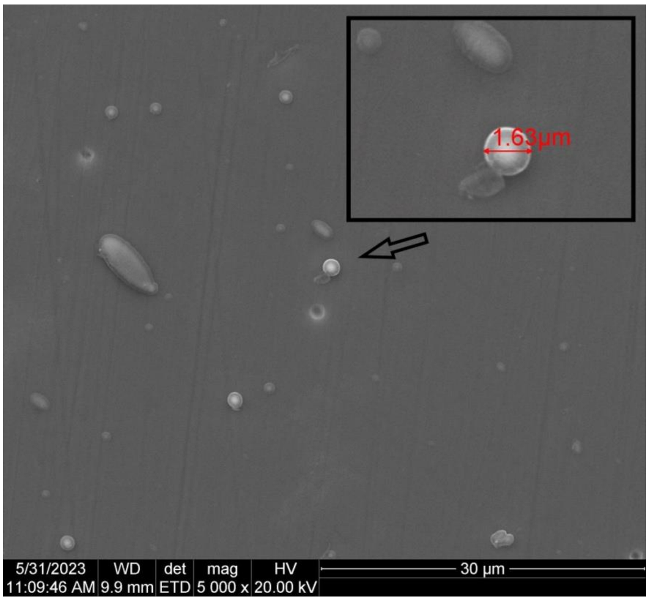

Fig. 1. SEM image of the surface of a 100Cr6 steel sample with a Ti−C:H coating

Chemical composition analysis (EDS) showed that the Ti−C:H coating consists of titanium and carbon, however, it was observed that the microdroplets have a different atomic ratio of carbon and titanium than the coating itself (Fig. 2). In the coating, 77.57 at. % of carbon and 22.43 at. % of titanium were recorded, while in the microdroplet 89.48 at. % of carbon and 10.42 at. % of titanium were recorded. It is to be supposed that the coating will have different tribological properties than microdroplets. They may have a particular impact in the first phase of the tribological test, because in the first phase of the running-in process, the detachment of microdroplets from the substrate can occur, which can introduce disturbances in the operation of the kinematic pair [8].

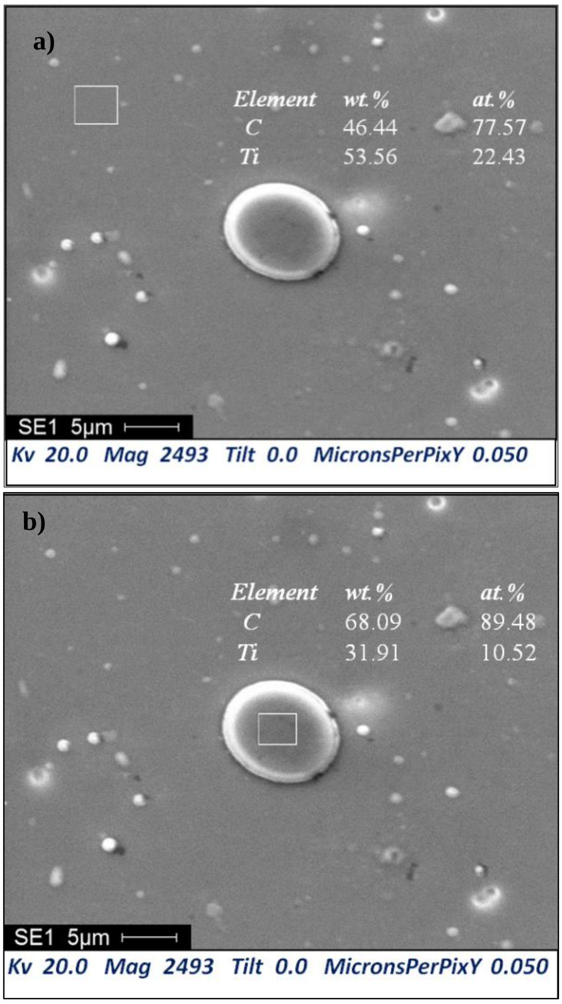

Fig. 2. EDS analysis of a Ti−C:H coating. a) coating, b) microdroplets

Thickness measurements performed using the Calotest method showed that the Ti−C:H coating deposited on 100Cr6 steel samples has an average thickness of 1.035 ±0.021 µm, so the process time was well chosen, since the assumption was to obtain a coating with a thickness close to 1 µm.

Roughness measurements showed that the roughness parameters increase after the Ti−C:H coating process, as shown in Table 2. The deterioration of these parameters is related to the presence of microdroplets on the surface of the coating, which were formed as a result of the coating process. These results confirm the conclusions of the observations made on the scanning electron microscope.

Table 2. Roughness parameters of uncoated samples and samples with a Ti−C:H coating

		Samples before the coating process	Samples after the DLC coating process
R_a	[nm]	5.3 ±0.57	6.3 ±0.57
R_z	[nm]	29 ±6.5	70 ±11
R_{max}	[nm]	48 ±29	85 ±17

Adhesion tests performed using the scratch test method on samples with applied Ti−C:H coatings showed that the first coating damage (L_{c1}) was recorded at a pressing force of 9 N, coating damage and tearing (L_{c2}) at a force of 16.32 N, and complete detachment of the coating from the substrate (L_{c3}) was recorded at a pressure of 40.15 N. The adhesion of the coating depends on, among other things, the process parameters of the coating deposition, stresses in the coating, its thickness, the hardness of the coating, and in the case of a-C:H coatings, the degree of hydrogenation [9, 11, 30]. The results of adhesion tests are shown in Fig. 3.

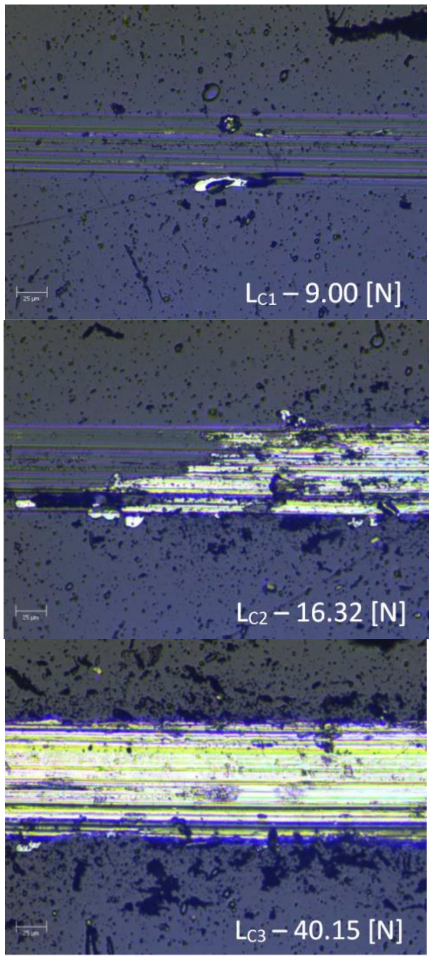

Fig. 3. Scratch test results of the Ti–C:H coating applied to a 100Cr6 steel substrate

Microhardness tests performed on a Fischerscope HM 2000 microhardness tester showed that the Ti−C:H coating applied to a 100Cr6 steel substrate has a higher hardness than the substrate, with a recorded value of 776.82 ±48.81 HV, and the substrate hardness is 58.93 ±0.6 HRC (~674 HV). This test was performed with a Vickers indenter depth of 0.25 μm to eliminate the influence of the substrate on the coating hardness measurements. The obtained hardness is due to the high degree of hydrogenation, since with its increase, the hardness of the produced coating decreases [11].

Tribological tests in a ball-on-disc configuration were carried out under dry friction conditions at ambient temperature. The tests were carried out for three kinematic pair variants. In the first variant, the Ti−C:H coating was applied to both the sample (disc) and the counter-sample (ball) made of 100Cr6 steel. The second variant is a sample coated with DLC and a counter-sample made of bearing steel without coating. In the third combination, samples made of steel were tested without the applied Ti−C:H coatings.

The recorded friction coefficient for all three kinematic pair variants indicates that the Ti−C:H coating significantly reduces the above-mentioned coefficient (Fig. 4), which is consistent with the data contained in the literature on DLC coatings [3, 5, 7, 25, 27, 38].

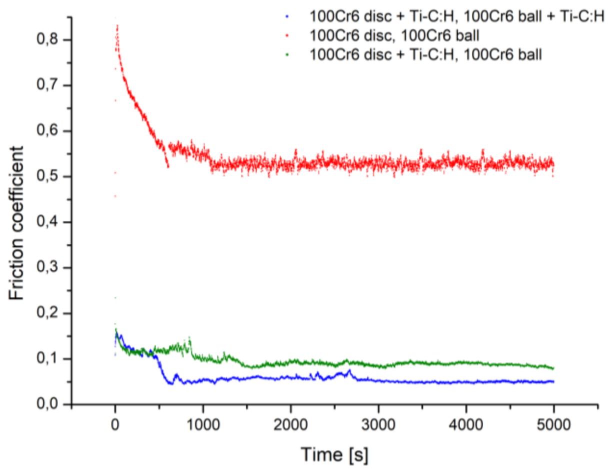

Fig. 4. Comparison of the recorded friction coefficient over time for all studied samples

In the first phase of the tribotest, the running-in process takes place in the kinematic pair, and then the recorded friction coefficient stabilizes. The calculated average value after 3000 s for each test indicates that the lowest friction coefficient was obtained for samples coated with Ti−C:H. The calculated values are presented in Table 3.

Table 3. Calculated friction coefficient after the break-in period of all studied samples

Sample	Friction coefficient
disk 100Cr6 + Ti−C:H, ball 100Cr6 + Ti−C:H	0.049 ±0.0014
disc 100Cr6 + Ti−C:H, ball 100Cr6	0.088 ±0.0035
disc 100Cr6, ball 100Cr6	0.540 ±0.0196

In the running-in period, in the case of the kinematic pair under study on which the Ti−C:H coating was applied, the recorded friction coefficient decreases (~500 s), which may be associated with the detachment and fall-off of microdroplets formed during the coating process, which is consistent with data found in the literature [8]. After the running-in period, the recorded friction coefficient decreases to a value close to 0.05. In the case of the kinematic pair in which only the disc was coated with the Ti−C:H coating, this process occurs more gently, and a sharp decrease in the friction coefficient resulting from the process of detachment of microdroplets from the coating was not observed.

Tribological tests have shown that the Ti−C:H doped DLC coating applied to both the sample and the counter-sample reduces the friction coefficient by eleven times compared to samples without coatings in dry friction conditions (0.049 vs 0.540). In the case of 100Cr6 steel samples with Ti−C:H coating and a counter-sample (ball) made of 100Cr6 steel, a friction coefficient six times lower than in the study on samples without coatings was recorded, and twice as high as in the case of the study for samples and counter-samples with DLC coatings.

The calculated wear rate of the sample and counter-sample (k_{vc}, k_{vb}) based on the conducted tests indicates that the lowest wear was recorded in the case of the pair on which the Ti−C:H coating was applied (Table 4).

Table 4. Wear rate of the sample and counter-sample after tribological testing

	Wear rate	
	Sample (disc)	Counter-sample (ball)
Sample	k_{vc} [mm^3/(N·m)]	k_{vb} [mm^3/(N·m)]
disc 100Cr6 + Ti−C:H, ball 100Cr6 + Ti−C:H	$1.86\cdot10^{-7} \pm6.29\cdot10^{-8}$	$3.03\cdot10^{-11} \pm7.3\cdot10^{-12}$
disc 100Cr6 + Ti−C:H, ball 100Cr6	$5.26\cdot10^{-7} \pm5.16\cdot10^{-8}$	$8.25\cdot10^{-9} \pm2.7\cdot10^{-9}$
disc 100Cr6, ball 100Cr6	$1.08\cdot10^{-5} \pm1.09\cdot10^{-6}$	$1.11\cdot10^{-5} \pm1.1\cdot10^{-7}$

The application of the Ti−C:H coating significantly reduces wear in the kinematic pair under study made of 100Cr6 steel compared to samples without coatings. In the case of the kinematic pair on which the Ti−C:H coating was applied only to the disc, it was observed that the wear is three times greater than in the case of the pair on which the coating was applied to both elements. The greatest changes in the case of the degree of wear were observed for the counter-sample (ball). It should be concluded that coating one element will effectively reduce the friction coefficient and wear of the sample under dry friction conditions, however, it is most effective to coat both elements in the kinematic pair.

The application of Ti–C:H coating can reduce the friction coefficient and wear rate, which allows for lower resistance and longer service life of kinematic pairs. However, their application is problematic due to limitations resulting from operation at elevated temperatures. Therefore, in order to reduce losses, it can be proposed to use them in every kinematic pair in internal combustion engines and drive systems, provided that they do not operate at elevated temperatures (above 100°C). An additional advantage is also the increased wear resistance in case of loss of lubrication due to failure or in boundary lubrication conditions.

3. Conclusions

Based on the conducted research, in which it was checked how the applied Ti−C:H coating would affect the reduction of the degree of wear and the friction coefficient, which is responsible for losses in the kinematic pair, the following conclusions were drawn:

– morphology studies of the surface performed on a scanning electron microscope showed that the coatings applied to the steel substrate did not show any defects (cracks, delamination, discontinuities, cracks), the only observation was the occurrence of microdroplets with of a size of several micrometers
– tribological testing showed that coatings applied to both the sample and the counter-sample reduced the friction coefficient by eleven times compared to samples without coatings under dry friction conditions (0.049 vs 0.540)
– analysis of the wear coefficients (k_{vc}, k_{vb}) of the sample and the counter-sample showed that DLC Ti−C:H coatings reduce wear by 58 times compared to samples without coating (k_{vc}), while in the case of the counter-sample (steel ball), the wear coefficient is several orders of magnitude smaller
– tribological tests performed on a sample made of 100Cr6 steel with a Ti−C:H coating and a counter-sample (ball) made of 100Cr6 steel, show a 6-fold lower friction coefficient than in the test on samples without coatings, and 2 times higher than in the case of the test for samples and counter-samples with DLC coatings
– coating one element with a Ti−C:H coating effectively reduces the friction coefficient and wear of samples in a kinematic pair under dry friction conditions, however, coating both elements in the kinematic pair is most effective
– the use of Ti−C:H coatings in kinematic pairs used in internal combustion engines and powertrains can reduce friction, especially in boundary lubrication or complete lack of lubrication (failure). This translates into reduced losses and wear, which is important in the search for solutions to reduce emissions of harmful compounds into the atmosphere, as well as to reduce fuel consumption
– taking into account the working conditions in the kinematic pairs of internal combustion engines and powertrains used in vehicles, the key is to select the right coatings, optimize them, and also seek solutions that primarily take into account the cooperation of the deposited coatings with lubricants
– to minimize losses generated in internal combustion engines and powertrains, it is essential to create a system in which individual kinematic pairs will have a reduced friction coefficient as a result of the methods of surface improvement used, strictly adapted to the conditions of their operation.

To find the most optimal solutions that lead to reduced losses, the continuation of research is planned:
– studies of kinematic pairs in which different PVD coatings will be applied to individual elements, and their optimization
– the influence of the lubricants used on the proposed PVD coatings that reduce wear and friction in kinematic pairs
– the influence of temperature on the generated friction coefficient in kinematic pairs coated with PVD coatings.

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