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THE EFFECT OF MeC NANOPARTICLES ON THE MICROMECHANICAL AND TRIBOLOGICAL PROPERTIES OF CARBON COMPOSITE COATINGS

WPŁYW NANOCZĄSTEK MeC NA WŁAŚCIWOŚCI MIKROMECHANICZNE I TRIBOLOGICZNE KOMPOZYTOWYCH POWŁOK WĘGLOWYCH

Key words: Carbon coatings, Nanocomposite, Low friction, Wear resistance.

Abstract Nanocomposite carbon coatings composed of a nanocrystalline phase and an amorphous carbon matrix (a-C or a-C:H) are an important group of coatings for tribological applications, especially if low friction is desired. Strong adhesion between the coating and the substrate as well as the ability to carry load are particularly important in ensuring the durability of the system. In this paper, the impact of a reinforcing phase in the form of hard carbides of chromium, titanium and tungsten (MeC) on the micromechanical and tribological properties of MeC/a-C coatings were analysed. The microhardness and modulus of elasticity using the indentation method and adhesion of these coatings to the substrate in scratch tests were determined. On the basis of tribological tests, the friction coefficient and wear rate of the coatings were determined during non-lubricated sliding contact with an alumina ball. The tested nanocomposite coatings showed very good sliding properties and wear resistance. The nc-WC/a-C and nc-TiC/a-C coatings exhibit the smallest coefficient of friction (below 0.1) and the highest wear resistance. The presence of nanocrystalline carbides in the amorphous carbon matrix limits the propagation of cracks in the coatings and allows the higher load carrying capacity.

Słowa kluczowe: powłoki węglowe, nanokompozyty, niskie tarcie, odporność na zużycie.

Streszczenie Nanokompozytowe powłoki węglowe złożone z nanokrystalicznej fazy i amorficznej osnowy węglowej (a-C lub a-C:H) stanowią ważną grupę powłok do zastosowań tribologicznych, zwłaszcza jeżeli pożądane jest niskie tarcie. Silna adhezja pomiędzy powłoką i podłożem oraz zdolność do przenoszenia obciążeń stykowych są szczególnie ważne w zapewnieniu trwałości systemu. W pracy dokonano analizy wpływu fazy wzmacniającej w postaci twardych węglików chromu, tytanu i wolframu (MeC) na właściwości mikromechaniczne i tribologiczne powłok MeC/a-C. Wyznaczono mikrotwardość i moduł sprężystości tych powłok metodą indentacyjną oraz ich adhezję do podłoża w teście zarysowania. Na podstawie badań tribologicznych w niesmarowanym styku ślizgowym typu kula/tarcza wyznaczono współczynnik tarcia oraz wskaźnik zużycia objętościowego powłok. Badane powłoki nanokompozytowe wykazały bardzo dobre właściwości ślizgowe oraz odporność na zużycie. Powłoki nc-WC/a-C i nc-TiC/a-C mają najmniejszy współczynnik tarcia poniżej wartości 0,1 i największą odporność na zużycie. Obecność w amorficznej osnowie węglowej nanokrystalicznych węglików ogranicza pękanie tych powłok i umożliwia przenoszenie większych obciążeń.

INTRODUCTION

Over the past two decades, significant developments in thin coating technology and the formation of their subsequent generations have been observed – from simple single TiN coatings to complex systems, such as multilayer, nanocomposite, or hybrid coatings **[L. 1]**. Nanocomposite and multilayer coatings are referred to as "third generation coatings." The technology of their production is well-known, but investigations into the direction of their structure and property modification are being conducted and, for this reason, the coatings can

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be used in new applications **[L. 2–5]**. A special group consisting of amorphous carbon a-C or hydrogenated amorphous carbon a-C:H coatings possess excellent sliding properties; however, their wear resistance is not too high due to their brittleness. The mechanical properties of carbon coatings depend strongly on the ratio of the sp^3 phase (typical for a carbon atomic arrangement in the crystalline lattice of a diamond) vs. the sp^2 phase (typical for a carbon atomic arrangement in the graphite plane). The content of individual fractions results from the method and production process conditions. With the increase in the sp^3 diamond-like phase, the hardness of the carbon coating increases, but so does its brittleness. Moreover, mainly because of high residual stress, the adhesion of the coating to the substrate is reduced. The wear resistance of carbon coatings can be improved, among others, by introducing transition metal atoms during the production process and the creation of nanocomposite structures (carbon matrix and hard carbides (MeC)) in this way. The nanocomposite coatings in tribological applications contain mostly nanocrystalline carbides of transition (d-block) metals, such as W, Ti, Cr, Ta, Zr, or Si, embedded in a soft matrix **[L. 6–8]**. Hard PVD coatings are able to work under very severe conditions when used, for example, as some anti-wear coatings for machining or plastic working tools. Highly loaded elements of the tribo-systems are exposed to intensive plastic deformation of the surface layers. Then, it is extremely important to simultaneously ensure sufficiently high hardness and ductility. The main parameter that characterizes this state is the hardness to elastic modulus H/E ratio. The H/E ratio, often referred to as elastic strain to failure, allows the qualification of the coating/substrate systems for the sake of their resistance to tribological (abrasive) wear **[L. 9–11]**. The hard carbon or ceramic coatings have the ability to carry heavy loads in point contact on the condition they have good support, i.e. when the coating is deposited on a hardened substrate. Duplex technology is one of the ways to increase the load capacity of the coating/substrate system, and thus improve the wear resistance in frictional contact **[L. 12]**. In this way, the materials tested in this work were produced.

In this paper, the effects of CrC, TiC, and WC carbide nanoparticles on the micromechanical and tribological properties of carbon composite coatings were analysed. In particular, the load-carrying ability of nc-MeC/a-C nanocomposite coatings as an important parameter in tribological contact was considered.

EXPERIMENTAL METHODS

The MeC/a-C nanocomposite coatings and a-C coating as a matrix deposited on face ends of ϕ 27×6 mm discs made of treated Ti-6Al-4V alloy has been examined. The investigated coating/substrate systems were obtained using duplex technology. In the first step, a Ti-6Al-4V alloy as a substrate was hardened by oxidation under plasma glow discharge for hard gradient layer preparation **[L. 13]**. The hardness and elastic modulus of the treated (Ti6Al4V (O2)) alloy were 10 GPa and 150 GPa, respectively. The coatings were fabricated by the reactive magnetron sputtering (RMS) technique.

The hardness and elastic modulus of the coatings and their adhesion to the substrate were investigated using a Micro-Combi-Tester (MCT) device from CSM Instruments. The hardness and elastic modulus were determined by the instrumental indentation method according to the Oliver and Pharr method. The three loads of 10, 20, and 50 mN were set on a Vickers diamond indenter. The indentation measurements were repeated ten times for each load, every time in a new area of the sample. The load carrying ability of the coating/substrate system was also investigated in indentation tests under a high load of 5 N. Coating adhesion to the substrate was examined by a standardized scratch test technique, according to the PN-EN ISO 20502:2016-05 standard. The scratch test enables the determination of several critical loads, such as the following: L_{C1} – causing cohesive cracks in the coating, L_{C2} – leading to the peeling of the coating – local exposure of the substrate (adhesive cracks), and L_{C3} – causing complete extensive delamination of the coating (extensive separation of the coating from the substrate on a large area considerably outside the scratch track). Three scratches were performed in a progressive mode using a Rockwell C indenter with the tip radius of 0.2 mm at the following test parameters: scratch length = 3 mm, maximum load = 30 N, and a relative speed of the indenter = 3 mm/min.

Tribological experiments were carried out in dry sliding contact with a 6 mm diameter Al_2O_3 ball (H = 19 GPa, E = 380 GPa). A typical ball-on-disc tribometer was used. Three repetitions for each sample were performed with the following test parameters: 5 N normal load, 0.05 m/s sliding speed, and 500 m sliding distance (20000 cycles on a 4 mm wear radius). The sample and counterpart surfaces were cleaned before each test and additionally, after setting in holders, were washed with an alcohol solution, and finally left to dry in an ambient atmosphere. The mating surfaces were analysed after friction using a light microscope (LM) and a Filmetrics Profilm 3D non-contact profilometer. The specific wear rate for the coating W_v (mm^3/Nm) was determined according to the following relationship (1).

$$W_v = \frac{V_{coating}}{F_n \cdot s} \tag{1}$$

where $V_{coating}$ is the wear volume of the coating calculated from the cross-sectional area of the wear track measured by a contact stylus profilometer (mm^3), F_n is the applied normal load (N), and s is the sliding distance (m).

RESULTS AND DISCUSSION

Characterization of the coating/substrate systems

The tested coatings were deposited by RMS on a hardened Ti-6Al-4V titanium alloy. The nano-composite coatings were composed of nanocrystalline carbides, such as CrC, TiC and WC, embedded in an amorphous a-C carbon matrix. The mean diameter of the CrC, TiC, and WC nanoparticles were determined as 3, 1.7, and 2.3 nm, respectively. MeC nanoparticles were uniformly distributed in composite coatings and the distance between them was in the range of 2 nm to 15 nm **[L. 7]**. In order to increase the adhesion of the coating to the substrate, a thin metallic (Cr, Ti, or W) intermediate layer was applied. The microstructure investigations showed that the nanocomposite coatings were dense, without any cracks or voids (**Fig. 1**). However, for the reinforced a-C coating, some discontinuities (micrometres) in the outer surface were observed (**Figs 5a, b**).

a) b) c)

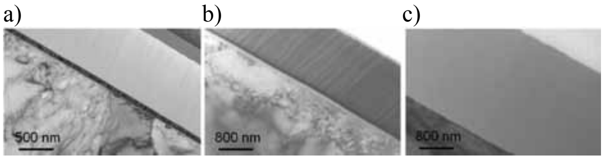

Fig. 1. TEM microstructure of the coatings: a) nc-CrC/a-C, b) nc-WC/a-C and c) nc-TiC/a-C on oxygen-hardened Ti6Al4V alloy

Rys. 1. Mikrostruktura powłok: a) nc-CrC/a-C, b) nc-WC/a-C and c) nc-TiC/a-C osadzonych na utwardzonym stopie Ti6Al4V (TEM)

Indentation tests

The hardness and elastic modulus of the tested coatings determined at indentation loads are shown in **Fig. 2**. The results of indentation measurements indicate a significant decrease in hardness as the load increases due to the effect of the substrate properties. Among the tested coatings, the highest hardness of 18 GPa and elastic modulus of 178 GPa are exhibited by the a-C coating. The nc-CrC/a-C composite coating has the lowest hardness, which was 10.4 GPa at a load of 10 mN and penetration depth of 159 nm.

Due to the load-carrying mechanism and stress concentration in the coating/substrate system, it is very important that the values of the elastic modulus of the coating and the substrate should be comparable. This relationship is particularly important in sliding point contact where the plastic deformation causes an increase in wear. Therefore, hardness is an insufficient parameter to describe the elastic behaviour of the coatings. In many research works, the hardness and elastic modulus ratio (H/E or H^3/E^2) is used as a parameter to better describe and predict the wear. The H/E ratio is associated with elastic strain to failure of the coating or the plasticity index (the coating ability to carry load), while H^3/E^2 is an indicator of the resistance to plastic deformation, and

a)

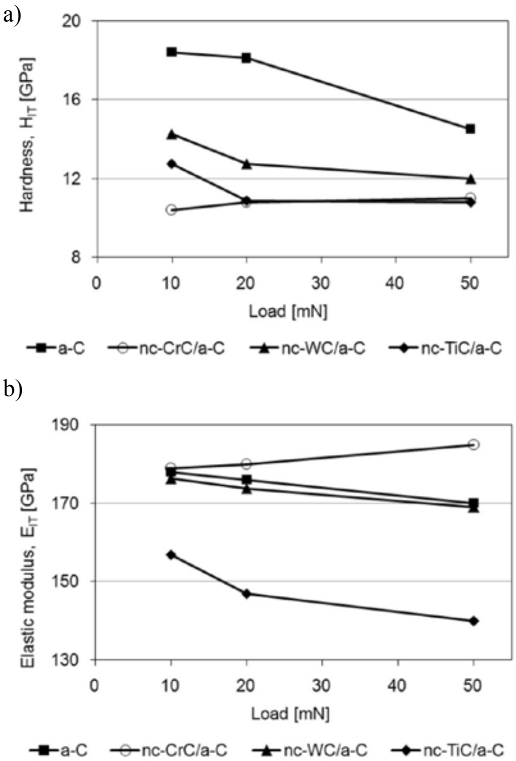

Fig. 2. Hardness H_{IT} and elastic modulus E_{IT} of the coatings at the load of the indenter 10, 20 and 50 mN

Rys. 2. Twardość H_{IT} i moduł sprężystości E_{IT} powłok przy obciążeniu wgłębnika 10, 20 i 50 mN

the increase in this ratio leads to an improvement in load capacity **[L. 14, 15]**.

In **Table 1**, the H_{IT}/E_{IT} and H_{IT}^3/E_{IT}^2 ratios calculated for the hardness and elastic modulus of the coatings measured at the load of 10 mN are shown. According to literature reports, the values of these ratios above 0.1 can bear high contact stresses. The a-C coating has the highest plasticity index; however, with a high concentration of stress, it is also characterized by high brittleness. While pressing the Vickers indenter with a load of 5 N, a distinct cracking of the tested coatings was observed (**Fig. 3**). The a-C and nc-CrC/a-C coatings show the most extensive cracks, including the detachment of the coating from the substrate. Initially, the cracks arise around the imprint, and the increase in load causes the development of cracks in the plane of the coating/substrate interface, and then delamination of the coating. However, for the nc-TiC/a-C and nc-WC/a-C coatings, characteristic frame-shaped cracks were observed only around the imprint. These nanocomposite coatings show much less destruction. There was no crushing of these coatings, despite the deformation of 5-6 micrometres on average, much more than the thickness of the coatings. The presence of MeC nanocrystallites in the amorphous carbon matrix as the reinforcing phase improves the cracking resistance of these coatings in accordance with the adopted general model **[L. 16]**.

Table 1. Hardness H_{IT} and elastic modulus E_{IT} at the load of the indenter 10 mN
Tabela 1. Twardość H_{IT} i moduł sprężystości E_{IT} przy obciążeniu wgłębnika 10 mN

Coating/substrate system	t [µm]	h_{max} [nm]	H_{IT} [GPa]	E_{IT} [GPa]	H_{IT}/E_{IT}	H_{IT}^3/E_{IT}^2 [GPa]
a-C on Ti6Al4V(O_2)	0.9	137 ±4	18.4 ±1	178 ±9	0.103	0.191
nc-CrC/a-C on Ti6Al4V(O_2)	0.9	159 ±10	10.4 ±1.9	179 ±11	0.061	0.036
nc-TiC/a-C on Ti6Al4V(O2)	3.1	174 ±9	12.8 ±1.4	157 ±13	0.082	0.085
nc-WC/a-C on Ti6Al4V(O2)	1.8	168 ±9	14.3 ±0.9	176 ±12	0.081	0.094

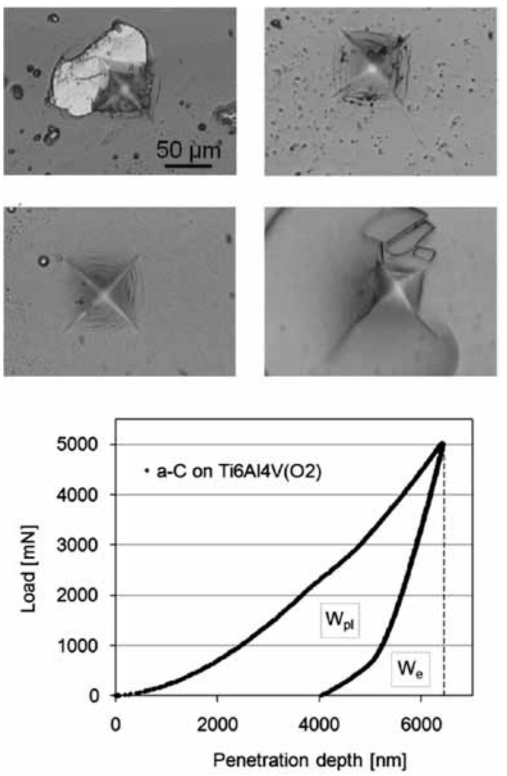

Fig. 3. Imprints of the coatings: a) a-C, b) nc-WC/a-C, c) ncTiC/a-C, and d) nc-CrC/a-C on Ti6Al4V(O2) alloy after indentation under 5 N, (LM, mag. 500x) and e) plot of a load-displacement curve obtained from indentation of a-C coating

Rys. 3. Obrazy odcisków po wciskaniu wgłębnika z obciążeniem 5 N powłok: a) a-C, b) nc-WC/a-C, c) ncTiC/a--C i d) nc-CrC/a-C na Ti6Al4V(O2), (LM, pow. 500x) oraz e) krzywa obciążenie-przemieszczenie podczas indentacji powłoki a-C

Figure 4 shows the contribution of plastic deformation work (W_{pl}) to the total work (W_{tot}) during the indentation of the tested systems. The W_{pl}/W_{tot} ratio was calculated on the basis of the load-displacement curves in the load range up to 50 mN (where $W_{tot} = W_{pl} + W_e$ and W_e is elastic deformation work (**Fig. 3**)). For nanocomposite coatings, the contribution of this work is much greater than for the a-C coating. This indicates the possibility of limiting the brittle cracking of such coatings, due to energy dissipation, by plasticizing the coating. Because of the different thickness of the tested coatings, the penetration depth was replaced with the

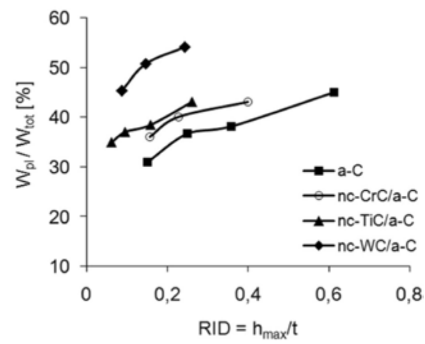

Fig. 4. Indentation plasticity work into total work ratio (W_{pl}/W_{tot}) as a function of relative indentation depth

Rys. 4. Udział pracy odkształcenia plastycznego względem całkowitej pracy podczas indentacji (W_{pl}/W_{tot}) w funkcji względnej głębokości penetracji

relative indentation depth (RID), i.e. the depth related to the thickness of the coating. At the lowest loads, the RID is at the level of 0.1, so the lack of influence of the substrate deformation on the measurements can be assumed. The increase in load leads to an increase in the W_{pl}/W_{tot} ratio, which is caused, to a large extent, by the plastic deformation of the substrate.

Scratch resistance of the coatings

The research results indicate that the nanocomposite coatings have significantly better scratch resistance in comparison with the a-C carbon coating. The nature of the composite coating destruction is similar. Initially, at the load of $L_{c1} = 6÷7$ N, there are arc-shaped cohesive fractures in the scratch track (**Fig. 5**). As the load increases, these cracks intensify and propagate beyond the scratch track. The load L_{c2} = 11 N caused the formation of adhesive cracks and the removal of large fragments of the nc-CrC/a-C coating on the edges and around the scratch track (**Fig. 5d**). The highest scratch resistance was demonstrated by the nc-TiC/a-C system on Ti6Al4V(O2), for which removal of small coating fragments was observed at an average load of 14 N (**Fig. 5h**). However, the unreinforced a-C coating completely peeled off, even at 7.7 N, and its delamination in a large area outside the scratch track also occurred (**Fig. 5b**). Similar values of the critical load around 19 N causing the destruction of the nc-TiC/a-C coating were found in the literature **[L. 17]**.

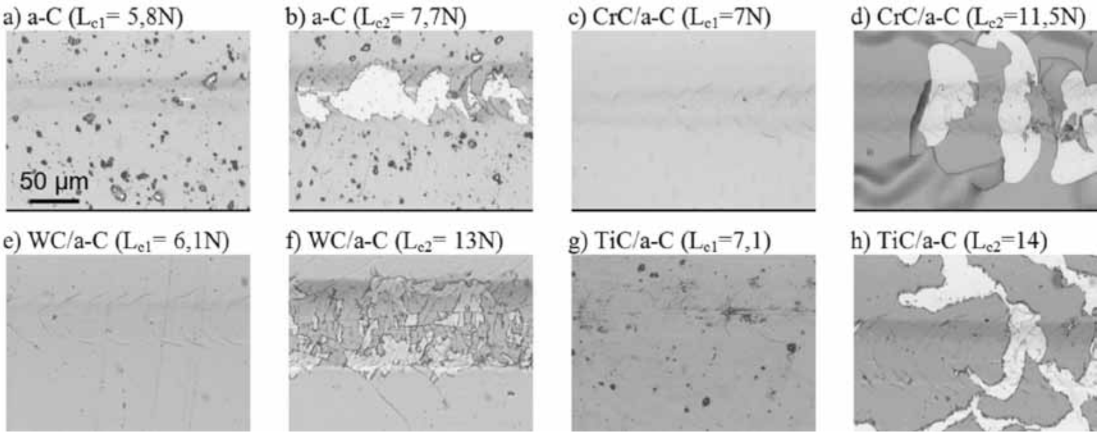

Fig. 5. Micrographs of the scratch track of the coatings on hardened Ti6Al4V(O2) alloy (LM, mag. x200)
Rys. 5. Obrazy toru zarysowania powłok na utwardzonym stopie Ti6Al4V(O2) (LM, pow. x200)

The scratch test is a commonly accepted method in assessing the adhesion strength of a coating to its substrate – a parameter decisive for the functional features of the coating/substrate system **[L. 18]**. Generally, for hard thin coatings, the microcracks are formed in the coating during scratching; at first under smaller L_{c1} load, and the development of these cracks induces the final adhesive failure at the higher L_{c2} load. Due to the usually different mechanical properties of the coating and substrate at their interface, there is a high concentration of shear stresses, which leads to adhesive destruction and coating delamination. Very hard coatings also exhibit very high stiffness and, for that reason, are easily fractured especially in point contact, and thus have insufficient wear resistance during friction. Therefore, the results of the scratch test also provide important information on the brittleness of the coating. In some works, the lower critical load L_{c1} is used as a parameter to indicate the cracking resistance of the coating **[L. 19]**, or some have even termed it "scratch toughness" **[L. 20]**. Zhang et al. **[L. 21]** pointed out that the coating toughness is proportional to both the lower critical load (L_{c1}) and the difference between the higher (L_{c2}) and the lower critical load, according to the following relationship:

$$CPR_S = L_{c1} (L_{c2} - L_{c1}) \quad (2)$$

The proposed parameter of CPR_S (Scratch Crack Propagation Resistance) has been used in the present work for a quick qualitative indication of the coating toughness (**Table 2**). However, it should be remembered that CPR_S is not a measure of toughness. CPR_S and the results of indentation tests at a 5 N load allow the coating's ability to carry load in sliding point contact to be determined.

Tribological properties

The nanocomposite tested coatings showed excellent sliding properties and very good wear resistance. During friction, the resistance to motion was gradually reduced mainly due to the creation of a self-lubricating layer on the sliding surfaces. A tribolayer was built from wear products of the coating (carbon particles) that were partly rubbing and dented on the surface of the sphere at the point of contact.

The wear of the ball was not observed, but a tribolayer formed on its surface, which is most often in a form of graphite-like structures. This behaviour was also observed for the a-C coating, however, only until the coating has been abraded to the substrate. Destruction of the a-C coating during friction occurred, on average, after 5000 cycles; therefore its wear rate was determined in this range. The tribolayer lubricated the frictional contact well, reducing movement resistance; thus, the coefficient of friction for the nc-WC/a-C, nc-TiC/a-C and a-C coatings reached the value below 0.1. Only for the nc-CrC/a-C coating was the friction coefficient greater, assuming a value of 0.2. This behaviour may be due to the more intense wear of the nc-CrC/a-C coating and the presence of the hard wear products in sliding contact.

Table 2. Critical load L_{c1}, L_{c2}, Scratch Crack Propagation Resistance (CPR_S) and specific wear rate (W_v)
Tabela 2. Obciążenie krytyczne L_{c1}, L_{c2}, odporność na propagację pęknięć przy zarysowaniu (CPR_S), wskaźnik zużycia (W_v)

Coating/substrate system	L_{c1} [N]	L_{c1} [N]	CPR_S	$W_v \cdot 10^{-6}$ [mm³/Nm]
a-C na Ti6Al4V(O_2)	5.8	7.7	11	0.89 ±0.1
nc-CrC/a-C na Ti6Al4V(O_2)	7	11.5	32	0.55 ±0.14
nc-TiC/a-C na Ti6Al4V(O2)	7.1	14	49	0.24 ±0.03
nc-WC/a-C na Ti6Al4V(O2)	6.1	13	42	0.08 ±0.02

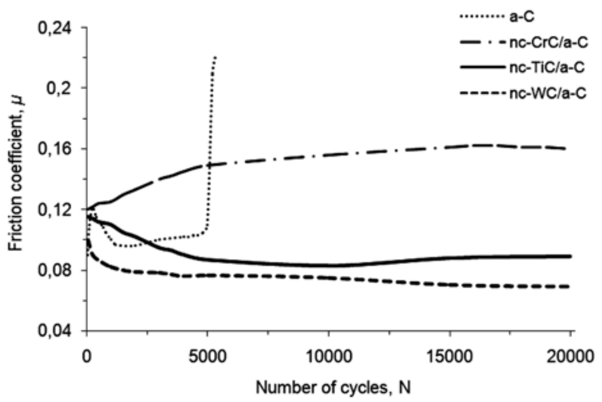

Fig. 6. Friction coefficient of the coatings in dry sliding contact against Al_2O_3 ball under load of 5 N
Rys. 6. Współczynnik tarcia powłok w styku ślizgowym z kulą Al_2O_3 przy obciążeniu 5 N

The tribolayer also separated the cooperating elements, so that their consumption was very small thanks to easy slide. Wear of the coatings was mainly abrasive and of a minimally adhesive nature when there was a transfer of the self-lubricating layer from the surface of the ball to the friction track, which was also observed in other works **[L. 17]**. In addition, due to the much higher brittleness of the a-C coating, it was additionally cracked. The tribological properties of the nc-TiC/a-C and nc-WC/a-C coatings were the best (**Table 2**) but also significantly different, despite their similar H/E and CPR_S values. The nc-WC/a-C wear rate was only $0.08 \cdot 10^{-6}$ mm^3/Nm and was three times lower than the wear rate of the nc-TiC/a-C coating with the largest CPR_S. However, the work of the plastic deformation W_{pl}/W_{tot} for the nc-WC/a-C coating is bigger, which indicates the possibility of limiting the brittle cracking of the coating, due to the energy dissipation while being plasticized. Destruction of the a-C coating can be explained in a similar manner. The a-C coating was not able to carry such a large load (average initial contact pressure p_m = 0.84 GPa) and was completely destroyed as a result of delamination. In addition, detailed observations of the friction track of the nc-TiC/a-C coating indicate the presence of hard particles to a larger amount than for the nc-WC/a-C coating, which intensified the wear process (**Fig. 7**). The surface roughness of the friction track of the nc-TiC/a-C coating was more than twice as high compared to nc-WC/a-C coating, and the Ra parameter reached the values of 0.01 and 0.004 μm, respectively.

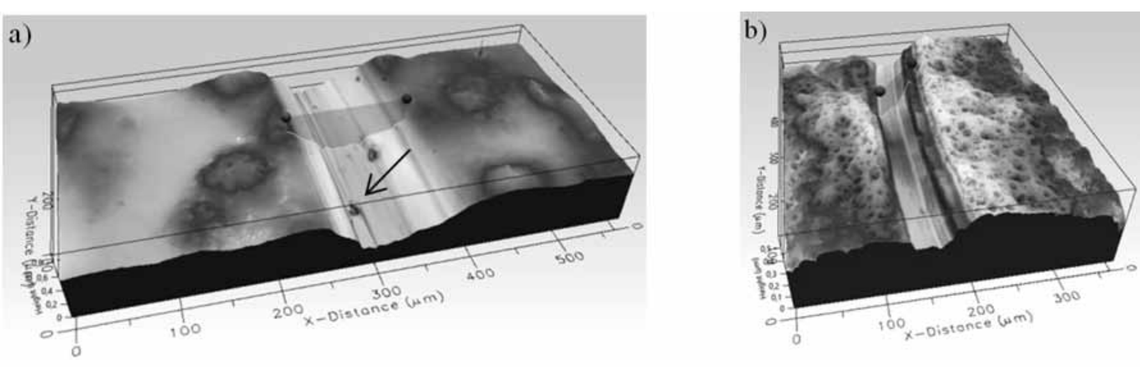

Fig. 7. 3D image of the wear track of the a) nc-TiC/a-C coating, and b) nc-WC/a-C coating after friction against Al_2O_3 ball
Rys. 7. Obraz 3D toru tarcia powłoki a) nc-TiC/a-C oraz b) nc-WC/a-C po współpracy z kulą Al_2O_3

SUMMARY

Amorphous carbon coatings produced by the reactive magnetron sputtering technique are distinguished by very good sliding properties, but their wear resistance in frictional point contact is not very high due to their brittleness. Despite the high ratio of H/E = 0.1, the a-C coating has the smallest plastic deformation (W_{pl}/W_{tot}) during indentation, which results in the easy propagation of brittle cracks. The introduction of chromium, titanium, and tungsten atoms in the carbon coating process deposition allowed the formation of nanocomposite coatings composed of the CrC, TiC, and WC nanocrystallites embedded in an amorphous a-C matrix. Nanocomposite coatings are characterized by significantly better scratch resistance and greater ability to carry contact stresses (the highest W_{pl}/W_{tot} values), and thus their resistance to wear increases. The results of tribological tests are correlated with the micromechanical properties of coatings. The best tribological properties were found for the nc-WC/a-C coating, for which the coefficient of friction was 0.09 and the wear ratio was only $0.08 \cdot 10^{-6}$ mm^3/Nm. Nanocrystalline MeC particles significantly increase the wear resistance of composite coatings compared to the unreinforced amorphous a-C matrix.

The hardness of the coating is an important parameter that only introduces preliminary information about its durability. In the interpretation of the test results of tribological properties, as well as in the prediction of the wear of thin hard coatings, it is extremely helpful to analyse the coating's resistance to cracking and load-carrying ability, which can be described by the parameters: H/E, CPR_S, and W_{pl}/W_{tot}.

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