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PVD coatings; DLC; wear resistance, adhesion

Krzysztof LUKASZKOWICZ
Division of Materials Processing Technology and Computer Techniques in Materials Science
Institute of Engineering Materials and Biomaterials
Silesian University of Technology
Konarskiego 18a, 44-100 Gliwice, Poland
Corresponding author. E-mail: krzysztof.lukaszkowicz@polsl.pl

COATINGS FOR TRANSPORT INDUSTRY

Summary. The investigations concerned structural analysis, as well as mechanical properties and wear resistant of MeN/DLC double-layer coating deposited by hybrid PVD/PACVD method. In sliding dry friction conditions, after the break-in time, the friction coefficient for the investigated elements is set in the range between 0.03-0.06.

POWŁOKI DLA PRZEMYSŁU TRANSPORTOWEGO

Streszczenie. Badania dotyczyły analizy strukturalnej oraz własności mechanicznych i odporności na zużycie ścierne dwuwarstwowych powłok typu MeN/DLC wytworzonych hybrydową metodą PVD/PACVD. W warunkach tarcia technicznie suchego po okresie docierania, zarejestrowany współczynnik tarcia dla badanych skojarzeń stabilizuje się w zakresie 0,03-0,06 w zależności od rodzaju powłoki.

1. INTRODUCTION

Tools, tooling components, and smaller engineered devices (i.e. shifter pins, fuel & brake system components, or seat belt retractor or track system parts, as well as, injection moulding, blow moulding, compression moulding, and forming tools) are very important parts in transport industry. Cars and aircraft have benefited from the development of nanomaterials production technologies and from better characterization tools and control of processes that are already widely established in industries (e.g. PVD and CVD processes for coatings) [1, 2].

In aerospace and automotive industries, aluminium and magnesium alloys are a major manufacturing activity. Nowadays, there is a worldwide demand for development of environmentally friendly and cost effective cutting or extrusion technologies including high-performance tool materials suitable for dry high-speed cutting or extrusion with high loads. Rapid advancements in many state-of-art industries, including plastic working, have depended mostly on the capabilities of surface engineering. Extrusion is a very popular process in plastic working, especially for non-ferrous metals and their alloys. The key user of such products is the aerospace and automotive sector [3-5].

In transport industries modern nanostructured coatings, for structural and functional applications, are used mainly for wear protection of machining tools and for the reduction of friction in sliding parts. DLC layers exhibit particularly advantageous tribological properties. Perfect and, owing to a variety of production techniques, differentiated properties of DLC layers have contributed to the intensive development of studies into their transport industrial applications [6, 7].

The aim of this paper is to examine the structure and tribological properties of the CrN+DLC, CrAlSiN+DLC, AlTiCrN+DLC, CrAlSiN+MoS_2 coatings deposited on the hot work tool steel.

2. INVESTIGATION METHODOLOGY

The production process of hybrid two-layer coatings of the hard nitride layer – low-friction layer DLC type was performed continuously with a π300 device by PLATIT fitted with LARC (Lateral Rotating Cathodes) cathodes and a CERC (Central Rotating Cathode) cathode in a single technological process. CrAlSiN/DLC, AlTiCrN/DLC, CrN/DLC and CrAlSiN/MoS_2 coatings were investigated. Main process parameters for substrate and coatings deposition conditions was presented in [8].

The fractographic tests of coatings were made on transverse fractures in a scanning electron microscope SUPRA 35 by ZEISS, fitted with the EDS chemical composition analysis system. Layers' surface topography tests and a fractional and multifraction analysis of the tested coatings were determined based on the measurements performed with an atomic force microscope (AFM) XE-100 by Park System.

The adhesion of the coatings to the substrate material was evaluated with a scratch test used commonly for coatings produced in physical vapour deposition processes.

The tests were made using a Revetest device by CSM using the following test conditions: pressing force range of 0-100 N; load increase rate (dL/dt) – 100 N/min; indenter movement rate (dx/dt) – 10 mm/min; acoustic emission detector sensitivity – 1.2. The character of the damage formed was assessed based on observations with an MEF 4A microscope by Leica.

A friction coefficient and the wear of coatings was determined in a test according to the ball-on-disk method. The tests were undertaken at room temperature with a T-01M device by ITE Radom under the following conditions: slide rate – 0.2 m/s (192 rpm); normal load – 19.62 N; friction radius – 10 mm; counter-specimen – an Al_2O_3 ball with 10 mm diameter; wear track – 1,000 m; ambient temperature – 23°C (±1°C); relative humidity – 30% (±5%); and at temperature increased with a high-temperature tribometer by CSM Instruments with the following conditions of the experiment: slide rate – 0.2 m/s (192 rpm); normal load – 5 N; wear radius – 5 mm; counter-specimen – an Al_2O_3 ball with 6 mm diameter; wear track – 500 m; ambient temperature - 400°C (±5°C).

The hardness tests of the deposited coatings were conducted with the Vickers method consisting of measuring the depth of indention that usually does not exceed the tenths of a micrometer, and the set pressure does not exceed 0.05 N, which eliminates the impact of the substrate material on coating hardness. Hardness identified this way, called dynamic hardness, determines a material's strength properties, including not only a plastic strain, but also an elastic strain. The measurements were made in the loading and unloading mode, where the indenter is loaded with the set force, the load is maintained for certain time, and then it is unloaded. A precision measuring system allows to record the depth of the indentation formed during loading, as well as during indenter unloading. A hardness test with the Vickers method was performed with nano-indenting made with a Shimadzu DUH 202 nanohardness tester.

3. DISCUSSION OF RESULTS

The fractographic tests made with the electron scanning microscope (Fig. 1) allows to assert that the tested coatings, depending on the applied system of layers, indicate a double-layer structure consisting of a hard nitride layer and a low-friction layer.

It was also found that a chromium-based transition layer exists well bound with a substrate that was fabricated to improve the coatings' adhesion to a hot-work tool steel substrate. The individual layers are deposited uniformly and tightly adhere to each other and to the substrate material. A morphology of the surface of fractures in the tested coatings is characterized by a compact structure.

The observations of the analysed coatings' surface topography using the atomic force microscopy (AFM) method revealed a varied surface topography of nitride and low-friction layers produced on the surface of hot-work tool steel.

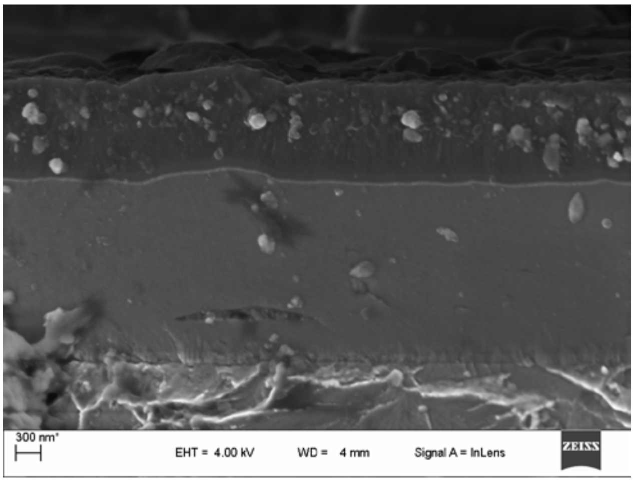

Fig. 1. Fracture image of CrAlSiN/DLC coating deposited onto the X40CrMoV5-1 steel substrate
Rys. 1. Powierzchnia przełomu powłoki CrAlSiN+DLC naniesionej na podłoże ze stali X40CrMoV5-1

Two types of morphology can be differentiated according to the type of layers. The first one occurs in the case of nitride layer produced by Physical Vapour Deposition with the arc method and is characterized by the existence of single droplet-shaped microparticles. The other one, though, exists for a low-friction DLC layer produced by Plasma-Assisted Chemical Vapour Deposition and is characterized by a high inhomogeneity relating to multiple droplet-shaped or nearly ball-shaped particles existing on the surface (Fig. 2).

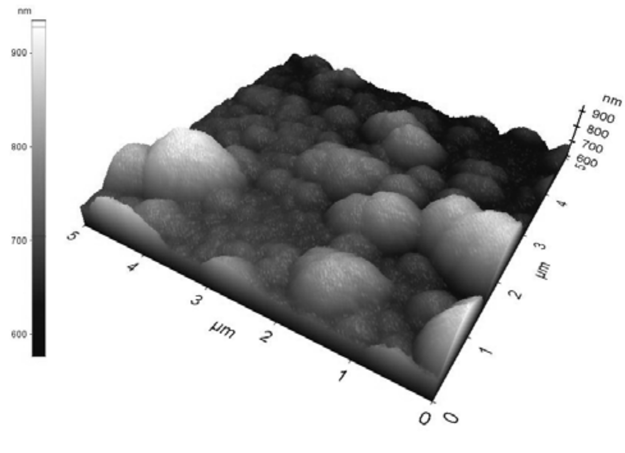

Fig. 2. CrAlSiN+DLC coating fracture surface (AFM)
Rys. 2. Topografia powierzchni powłoki CrAlSiN+DLC

Coating adhesion of the substrate material is one of the crucial concepts concerning the deposition of hard ceramic material onto substrate. Hence adhesion is one of the most important properties of coatings produced with physical and chemical vapour deposition. If adhesion is inappropriate, the whole functionality of a coating may be compromised.

The critical load L_{C1} and L_{C2} values were determined with a scratch test with a growing load allowing to determine the values of the force causing coating damages. The load at which the first coating damages occur is termed in the literature as the first critical load L_{C1}. A value of the first critical load is related to cohesion damages connected with the chipping of a material inside the coating, without exposing (uncovering) the substrate material. The second critical load L_{C2} is characterised by the total destruction of a coating. An aggregate list of the tests' results is presented in Tab. 1. The highest values of the critical load L_{C1} and L_{C2} account for, respectively, 36 and 76 N, and,

therefore, the best coating adhesion to the substrate was achieved for CrAlSiN+DLC coatings. The other critical load values measured, signifying coating adhesion to the substrate, do not exceed 70 N.
A critical load registered during an adhesion test does not depend only on mechanical strength (adhesion, cohesion) of the coating-substrate system but also on various properties related to the coating and substrate material, such as: substrate hardness and roughness, coating hardness and roughness, coating thickness, friction coefficient between a coating and indenter, internal stresses in the coating as well as the conditions of the test itself. In general, the first symptoms of a damaged coating in the majority of the tested coatings are represented by arch-like cracks caused by stretching and chipping occurring at the bottom of the scratch formed during a scratch test. In few cases, minor chipping occurs at the scratch peripheries.

Table 1

Aggregate list of mechanical properties

Type of coatings	Hardness HV	Critical load L_{C1}, N	Critical load L_{C2}, N
CrAlSiN/DLC	3846±471/1980±160	36±4	76±6
AlTiCrN/DLC	3384±359/1659±92	28±3	67±8
CrN/DLC	2442±235/1894±105	18±3	46±4
CrAlSiN/MoS_2	3798±569/1086±63	19±2	63±2

An abrasive wear resistance test in dry slide friction conditions with the ball-on-disk method at room temperature was performed to determine the tribological properties of the tested coatings deposited on a hot-work X40CrMoV5-1 tool steel substrate. Fig. 3 illustrates the diagrams of changes in a dry friction coefficient μ obtained during tests of wear in relation to an Al_2O_3 counterpart at the temperature of 20°C for a wear track of 1000 m.

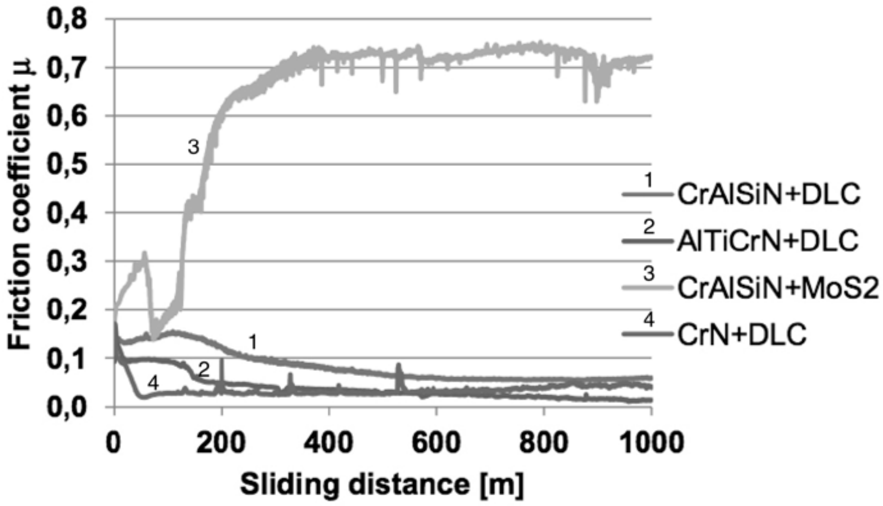

Fig. 3. Relationship between the friction coefficient and wear track obtained based on a wear resistance tests with the ball-on-disk method for the coatings analysed at the temperature of 20°C

Rys. 3. Zależność współczynnika tarcia od drogi tarcia uzyskana na podstawie badania odporności na ścieranie metodą ball-on-disk dla analizowanych powłok w temperaturze 20°C

Nearly all the friction curves for coatings with a low-friction DLC layer have a similar characteristic, with the initial transient state with an unstabilized curve, during which a friction coefficient decreases as the wear track increases until the set state is reached, which usually takes place after approx. 100-200 m. The registered friction coefficient for the examined combinations stabilizes within 0.03-0.06 according to the coating type (Tab. 2) in the conditions of dry technical friction, after the running-in period.

Table 2

Aggregate list of tests results of abrasive wear at temperature of 20°C

Type of coatings	Thickness, μm	Friction coefficient μ	Wear rate of coatings k_{vc}, mm^3/Nm	Wear rate of counterpart k_{vb}, mm^3/Nm
CrAlSiN/DLC	2,0/1,3	0,06	$4{,}54\times10^{-7}$	$9{,}71\times10^{-9}$
AlTiCrN/DLC	1,2/1,9	0,03	$3{,}30\times10^{-7}$	$4{,}53\times10^{-9}$
CrN/DLC	1,1/0,5	0,04	$1{,}58\times10^{-7}$	$6{,}56\times10^{-9}$
CrAlSiN+MoS_2	2,0/0,3	0,15/0,72	$2{,}22\times10^{-6}$	$4{,}83\times10^{-8}$

The coatings were not worn through entirely in any of the cases as the maximum depths of wear are smaller than their thickness. The visible build-ups on the wear surfaces of the tested specimens may influence deviations in the friction coefficient on the charts recorded during wear tests.

It can be assumed based on the observations of traces of coatings' wear made with a confocal microscope that relative displacement of the material of the coating and counterpart occurs during friction. The layers formed are characterised by high hardness (Tab. 1) and resistance to abrasive wear (Tab. 2) resulting in a narrow and shallow wear track in all the cases.

The coatings and counterpart examined exhibit high tribological properties. The values of the coating K_c and counterpart K_b wear coefficients were recorded at the level of 10^{-7} mm^3/Nm and 10^{-9} mm^3/Nm (Tab. 2). For a CrAlSiN+MoS_2 coating, however, where the majority of build-ups existed, the highest μ friction coefficient was noticed and most intensive counterpart wear. A friction coefficient μ for AlTiCrN+DLC, CrAlSiN+DLC and CrN+DLC coatings and a value of K_c and K_b indicators are lower, accordingly, evidencing their high resistance to abrasive wear.

Fig. 4 illustrates the diagrams of changes in the dry friction coefficient μ obtained during the tests of wear in relation to a Al_2O_3 counterspecimen at the temperature of 400°C for a wear track of 500 m for a load of 5 N. The lowest value of the K_c coating and K_b specimen wear indicators (Tab. 3) were noted for a CrN+DLC coating which confirms its high wear resistance at elevated temperature and is consistent with the results of operational tests performed at 400°C.

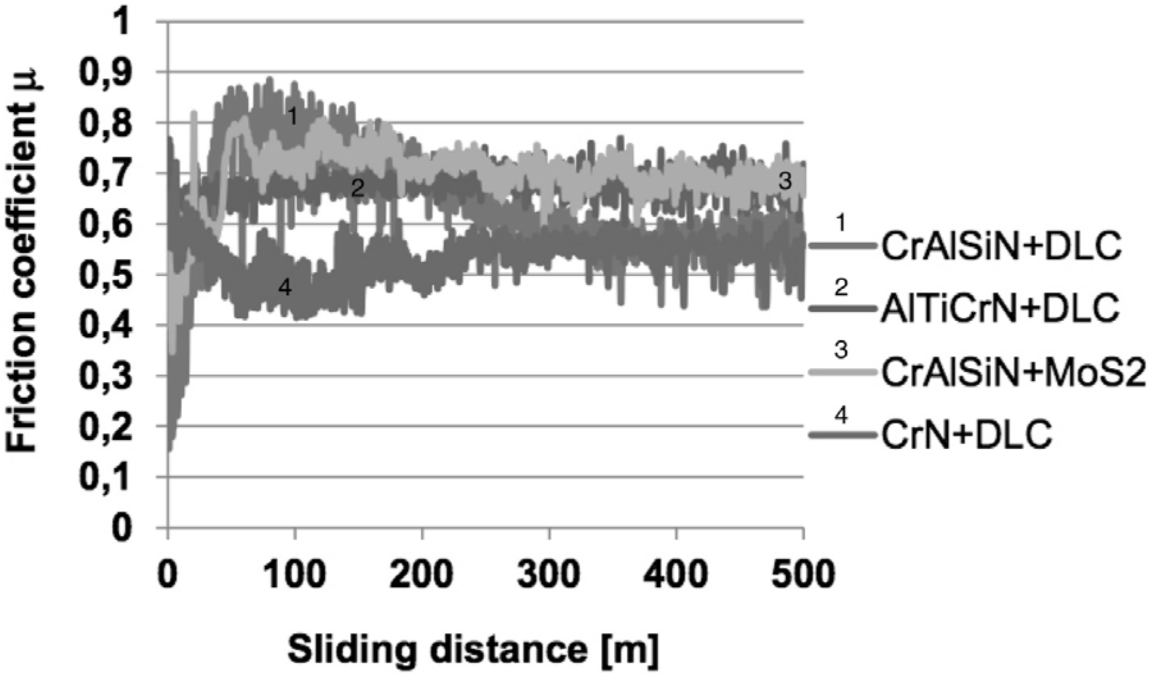

Fig. 4. Relationship between the friction coefficient and the wear track obtained based on a wear resistance test with the ball-on-disk method for the coatings analysed at the temperature of 400°C

Rys. 4. Zależność współczynnika tarcia od drogi tarcia uzyskana na podstawie badania odporności na zużycie ścierne metodą ball-on-disc dla analizowanych powłok w temperaturze 400°C

Table 3

Aggregate list of tests results of abrasive wear at temperature of 400°C

Type of coatings	Friction coefficient μ	Wear rate of coatings k_{vc}, mm^3/Nm	Wear rate of counterpart k_{vb}, mm^3/Nm
CrAlSiN/DLC	0,56	$4{,}70\times10^{-6}$	$2{,}73\times10^{-7}$
AlTiCrN/DLC	0,67	$8{,}69\times10^{-6}$	$6{,}60\times10^{-7}$
CrN/DLC	0,54	$4{,}35\times10^{-6}$	$2{,}46\times10^{-7}$
CrAlSiN+MoS_2	0,69	$1{,}88\times10^{-5}$	$3{,}96\times10^{-7}$

4. SUMMARY

The destruction mechanisms existing mainly on the tools, tooling components, and smaller engineered devices surfaces can be more broadly analysed and identified accurately through interpreting adequately mutual dependencies between the surface layer and substrate properties and structure and the external factors. The investigated coatings with DLC top layer produced by hybrid process, were properly deposited on the X40CrMoV5-1 hot work tool steel substrate. It was found after carrying out the ball-on-disk tests that coatings with a low-friction DLC layer exhibit the smallest friction coefficient. The coating adhesion scratch tests disclose the cohesion and adhesion properties of the coatings tested. In virtue of the tests carried out, it was found that the critical load L_{C2} fitted within the range 46-76 N for the coatings deposited on a substrate made of hot work tool steel. An abrasive wear resistance test with the ball-on-disk method was performed to fully determine the functional and operating characteristic of the analysed coatings. It was found after carrying out the test that coatings with a low-friction DLC layer exhibit the smallest friction coefficient.

References

1. Vetter, J. & Barbezat, G. & Crummenauer, J. & et. al. Surface treatment selection for automotive applications. *Surface & Coatings Technology*. 2005. Vol. 200. No. 5-6. P. 1962-1968.
2. Baragetti, S. Fatigue resistance of steel and titanium PVD coated spur gears. *International Journal of Fatigue*. 2007. Vol. 29. No. 9-11. P. 1893-1903.
3. Hovsepian, P.E. & Luo, Q. & Robinson, G. & et. al. TiAlN/VN superlattice structured coatings: A new alternative in machining of aluminium alloys for aerospace and automotive components. *Surface & Coatings Technology*. 2006. Vol. 201. P. 265-272.
4. Tański, T. & Labisz, K. & Lukaszkowicz, K. Structure and properties of diamond-like carbon coatings deposited on non-ferrous alloys substrate. *Solid State Phenomena*. 2013. Vol. 199. P. 170-175.
5. Altun, H. & Sen, S. The effect of DC magnetron sputtering AlN coatings on the corrosion behavior of magnesium alloys. *Surface & Coatings Technology*. 2005. Vol. 197. No. 2-3. P. 193-200.
6. Bobzin, K. & Bagcivan, N. & Goebbels, N. & et. al. Lubricated PVD CrAlN and WC/C coatings for automotive applications. *Surface & Coatings Technology*. 2009. Vol. 204. No. 6-7. P. 1097-1101.
7. Lukaszkowicz, K. & Kriz, A. & Sondor, J. Structure and adhesion of thin coatings deposited by PVD technology on the X6CrNiMoTi17-12-2 and X40CrMoV5-1 steel substrates. *Archives of Materials Science and Engineering*. 2011. Vol. 51. P. 40-47.
8. Lukaszkowicz, K. Forming the structure and properties of hybrid coatings on reversible rotating extrusion dies. *Journal of Achievements in Materials and Manufacturing Engineering*. 2012. Vol. 55. P. 159-224.

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