ANALYSIS OF TRIBOLOGICAL PROPERTIES OF MULTI- AND NANOCOMPOSITE COATINGS DEPOSITED ON A PM SUBSTRATE

D. Jakubéczyová, B. Ballóková

Abstract
The evaluation of tribological properties of PVD multi- and nanocomposite coatings on the PM substrate Böhler S390 is presented. Tribological properties were evaluated by the ball-on-disc test and by metallographical analysis of the wear track after testing at room temperature. Coefficient of friction and specific wear rates were evaluated. Damage mechanisms were identified and their relationship with structural characteristics was inferred.
PVD technology is among the modern ways of depositing thin coatings onto the surfaces of machine parts. Technological methods of deposition process of thin coatings are constantly innovated. At the same time, new types of coatings are being developed. The evaluation of structure, composition and properties of coatings is necessarily all development time. An AlTiNmulti and nanocomposite nACo (TiAlSiN) coatings are used to increase the service-life of cutting tools. Because of this, it is important to analyse the behaviour of coated materials in a tribological environment.
Keywords: PM substrate, coating, multilayer, nanocomposite, wear, tribology

INTRODUCTION

Modern nanostructured coatings, intended for structural and functional applications, are used mainly for wear protection of machine tools and for the reduction of friction in sliding parts. The nanocomposite coatings are increasingly used in a number of applications involving tools for farming, e.g. aluminum extrusions. The demands on the tool material of an extrusion die are high hardness, creep resistance, yield strength, toughness at elevated temperatures, wear and corrosion resistance [1]. Nanostructural and particularly nanocomposite coatings, deposited by physical or chemical vapour deposition, are considered to be very interesting premium technologies for protection and modification of product surfaces. They enable one to obtain materials with unique physical and chemical properties, e.g. extremely high indentation hardness (40-80 GPa) [2,3], corrosion resistance [4], excellent high temperature oxidization resistance [5], as well high abrasion and erosion resistance [6,7]. The multilayers, having nanometer-scale-thickness layers, exhibit many new structural characteristics and improved mechanical and industrial properties of tools and parts. Polycrystalline multilayers possess zones of varying density that generally result in increased hardness and better resistance to wear. The use of materials with good tribological properties of individual layers within multi- and nanolayers opens up a number of possibilities for development of new hard coatings for industrial use. Nanocomposite

Dagmar Jakubéczyová, Beáta Ballóková
Institute of Materials Research, Slovak Academy of Science, Watsonova 47, Košice, Slovak Republic

structure with nanocrystalline (nc-) grains embedded in an amorphous (a-) matrix improves considerably the properties such as high nanohardness, high thermal resistance, high resistance to wear and oxidization.

EXPERIMENTAL DETAILS

Coatings were deposited onto the substrate produced by powder metallurgy (PM) HSS S390 (Böhler) in the form of discs with dimensions of Ø 30 mm × 6 mm. Multilayer AlTiN was deposited by the system ARC PL 1000, equipped with four cathodes, and a nc-$Ti_{1-x}Al_xN$)/a-Si_3N_4 (nACo) by the machine LARC π-80 (LARC®-LAteral Rotating Arc Cathodes) with two cathodes [8]. After thermal processing to the hardness of 64 HRC and 1280 HV0.5, the structure of the PM substrate consisted of tempered martensite and carbides. Thickness of the deposited layers was determined by Calotest and reached 2.63 µm for AlTiNmulti layer and 2.22 µm for nACo layer. The method is described in detail in [9].

The roughness of the layers was tested by a profilometer Hommel Tester T 1000 and achieved Ra = 0.09 µm for AlTiNmulti and Ra = 0.07 µm for nACo.

The microhardness of the substrate and the coatings was measured by a Leco Micro Hardness Tester, and after coating, microhardness HV0.5 of both coatings was higher in comparison with the substrate. The nanocomposite nACo achieved a higher microhardness, namely 2510 HV0.5, than the AlTiNmulti, which reached microhardness of 2090 HV0.5.

Tribological properties of coated specimens were tested by HT Tribometer CSM using a ball-on-disc test device (WC ball). The test conditions are presented in Table 1. The wear tracks were examined by a light microscope Olympus GX 71.

Tab.1. Parameters of ball-on-disc test.

Pin ball	WC - ϕ 6 mm
Speed of ball *v*	4 $cm{\cdot}s^{-1}$
Number of cycles *N*	10 000
Track radius *r*	2, 4, 6, 8 mm
Load *L*	5 N
Temperature	20°C

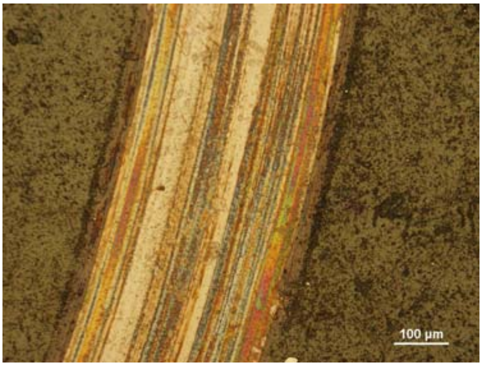
The depth and shape of the wear tracks were measured by a confocal microscope Plu neox 3D Profiler by Sensofar. The average through cross-section area was calculated and subsequently the volume of the removed material was estimated. The specific wear rate (*w*) was then expressed according to the standard ISO 20808, 2004 [10], as the volume loss $V\ (mm^3)$ per distance $L\ (m)$ and the applied load $F_p\ (N)$:

$$w = \frac{V}{L.F_p} \qquad (1),$$

and was used for comparison purposes.

RESULTS AND DISCUSSION

After determination of the described properties, the specimens were subjected to tribological testing by a WC ball at 20°C. Figures 1 and 2 illustrate morphology of wear tracks at radiuses r = 2 mm and 8 mm and wear distances of 126 and 502 m for both coatings. In the case of the AlTiNmulti coating, the increase in wear distance resulted in an almost 1.5-fold increase in wear track width. Such tendency was not observed for the nACo coating, which showed only a slight change in track width, as presented in Table 2. In all the tracks analysed we failed to identify any significant failure of coatings (detachment, tearing, etc.). Visible laminar structure of the wear track with individual layers of nanometer dimensions could be observed.



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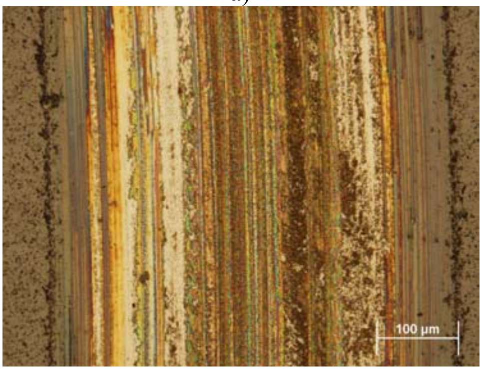

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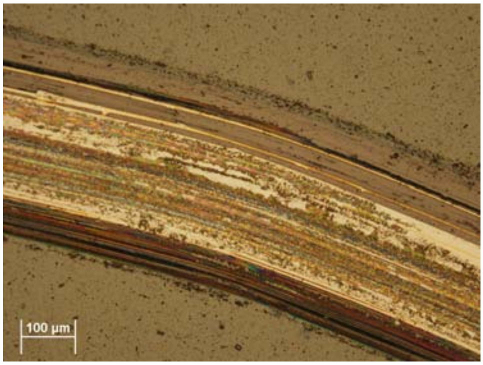
Fig.1. The morphology of the wear track of AlTiNmulti with a radius a) r = 2 mm and b) r = 8 mm, LM.



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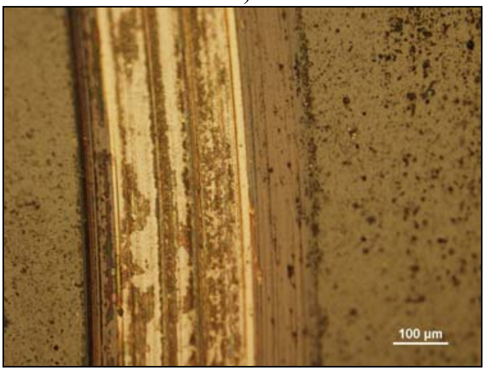

b)

Fig.2. The morphology of the wear track of nACo with a radius a) r = 2 mm and b) r = 8 mm, LM.

The results of measurements of the wear track depth performed by the confocal microscope for both materials enabled us to state that under the given test conditions, the depth of the wear track did not exceed the coating thickness (2.22 µm or 2.63 µm) and did not extend to the base layer (substrate). The maximum depth of the tribo-track in AlTiNmulti coating was 1.3538 µm and in the nACo nanocomposite layer it reached 1.340 µm, as seen in Table 2. Figures 3 and 4 show the cross sections of coatings observed by scanning electron microscopy (SEM) and elements detected by LineScan EDS analysis based intensity of respective signals. In both coatings, there is a surface AlN layer resistant to thermal-oxidation. In the AlTiNmulti coating it is the TiN layer, deposited as the first, due to its excellent adhesion to the substrate. The nanocomposite nACo coating contains metal nitrides AlN and TiN embedded in an amorphous matrix Si_3N_4, as indicated by the distribution of elements in Fig.4.

Tab.2. The results of measurements obtained for wear tracks after tribological tests by confocal microscopy.

Experimental materials	Track radius [mm]	Wear distance [m]	Depth of wear track [µm]	Track width [µm]
AlTiNmulti/S390	2	126	1.2252	363.820
	4	251	1.3538	401.258
	6	377	1.0769	429.596
	8	502	0.9055	457.48
nACo/S390	2	126	1.3306	272.903
	4	251	1.3235	302.258
	6	377	1.2764	281.207
	8	502	1.3400	309.506

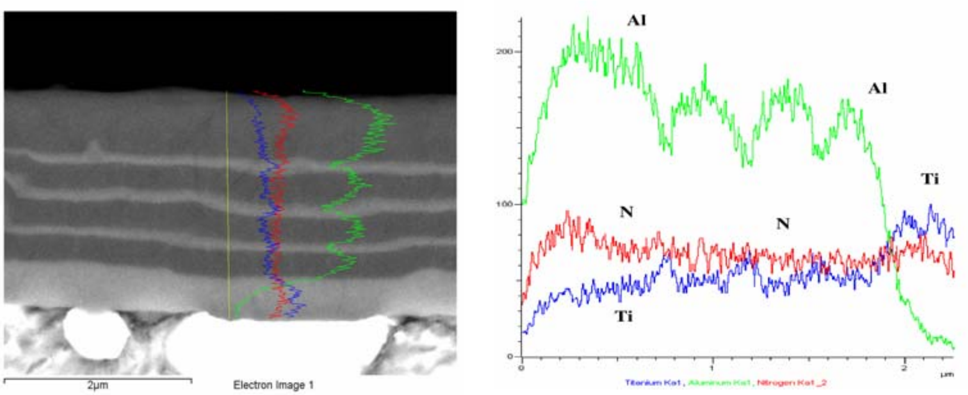

Fig.3. Cross-section of the coating AlTiNmulti and LineScan EDS analysis, SEM.

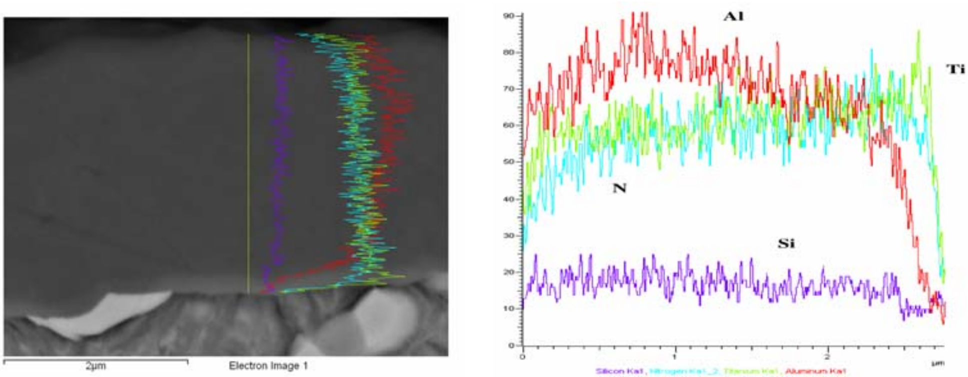

Fig.4. Cross-section of the coating nACo and LineScan EDS analysis, SEM.

The relationship between the coefficient of friction (COF) and the wear distance after the pin-on-disc test at ambient temperature is shown in Fig.5. Figure 6 illustrates the dependence of the specific wear rate on the wear distance.

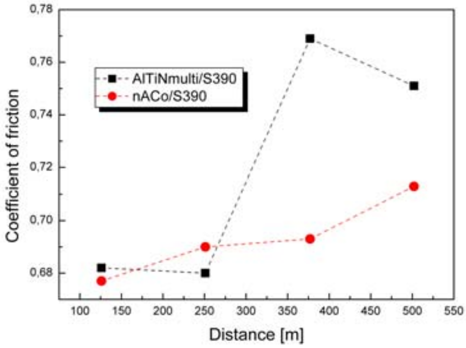

Fig.5. Dependence of the friction coefficient on the wear track distance.

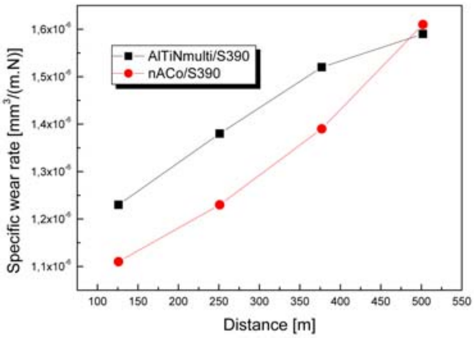

Fig.6. Dependence of the specific wear rate on the wear track distance.

With both coatings, the COF increased with the lengthening distance of wear. Less pronounced changes in COF were observed at shorter distances up to 250 m, and the COF values for AlTiNmulti coating fluctuated around 0.68. At distances above 300 m, the COF increased considerably and reached 0.77 for the AlTiNmulti coating. A similar increase in COF level was also observed for nACo coating, but a marked increase in COF for this coating was observed at distances above 400 m and the value of 0.70 was reached. We anticipated the lower level of COF in the nACo coating due to interaction of materials which come into contact: the WC ball and the hard nanocomposite nACo coating.

The dependence of the specific rate of wear on wear distance indicates that for both coatings the increased wear distance results in an increased specific wear rate. A more pronounced increase is observed with AlTiNmulti coating, reaching almost 10 - 14% at test distances up to 400 m. With the lengthening of the wear track of material coated nACo, an additional increase in wear rate occurs up to the level of $1.6x10^{-6}$ $mm^3/m\cdot N$ at 502 m wear distance. Such tendency of increasing the *w* level does not apply to materials with AlTiNmulti coating, as at wear distances exceeding 400 m, the increase in the specific wear rates is not as pronounced. At the distance of 502 m, the values of the specific wear rates become almost equal for both coatings and the coating wear rate reaches ~$1.6x10^{-6}$ $mm^3/m\cdot N$.

The study [11] showed that lower hardness resulted in better resistance to wear. During the test the coating is stressed, undergoes slow wear and, due to its relatively low hardness, acts as a solid lubricant. In our study the hardness of tribological pairs coating - ball/pin differed (WC-ball; 17-24 GPa [12]). Nanometric harder fragments of coating are forced into micropores of the WC ball and act as an effective „grinding tool". Intensity of friction reduction of such ball during the test is indirectly proportional to its relative velocity on the coating and, gradually, the intensity of the ball reduction and its velocity on the surface become equal. Under such conditions, the grinding effect of the pin on the surface of the coating is suppressed, coefficient of friction decreases and a sliding layer is produced.

CONCLUSIONS

Attempts were made to evaluate tribological properties of multi- and nanocomposite thin films. Comparison of AlTiNmulti and nanocomposite nACo coatings did not fulfil our expectations; we expected better tribological properties of the nACo coating. Our anticipations were not fulfilled, or the employed method of testing of the respective coatings was only one of many that enable one to obtain additional characteristics important for determination of quality of deposited coatings.

- Tribological properties were determined by the ball-on-disc test and the friction coefficient and its changes during the test were investigated. The wear character of wear tracks after testing at 20°C was evaluated and documented by light, confocal and scanning electron microscopy. Behaviour of the nanocomposite nACo was better than that of AlTiNmulti coating, as shown by morphological assessment of the wear track.
- The deposited types of coatings resulted in increased hardness of the base material. The substrate-coating interface was without relevant failures, which confirmed adequate adhesion properties of the system.

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