DETERMINATION OF TRIBOLOGICAL PROPERTIES OF MULTILAYER COATINGS BASED ON NITRIDES

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The coefficient of friction (COF) of AlTiCrN and AlTiN multilayer coatings is evaluated. The results of research on the evaluation of the COF, the values of the removed material volume and the specific abrasion values for multilayer coatings based on nitrides for different path radius are presented. Various wear mechanisms of worn surfaces for the different loading conditions at a temperature of 400°C are found. It is shown that the AlTiN coating can be used in environments with higher temperatures, i.e. also on the inserts of molds used in high-pressure casting of Al and its alloys.

Keywords: *surface engineering, coatings, friction, nitrides, tribology.*

Подано результати оцінювання коефіцієнтів тертя, об’єму видаленого матеріалу та питомого зношування для багатошарових покриттів на основі нітридів AlTiCrN і AlTiN за різних радіусів траєкторій тертя. Виявлено неоднакові механізми тертя поверхні зношування за різних умов навантаження при 400°C. Виявлено, що покриття AlTiN можна використовувати у високотемпературних середовищах, тобто на вставках пресформ за високотемпературного литва алюмінію та його сплавів.

Ключові слова: *інженерія поверхні, покриття, тертя, нітриди, трибологія.*

Introduction. Die casting molds are subjected to various thermal and mechanical loads. Generally, heat cracking, die soldering and erosion are some of the most relevant phenomena that reduce dies lifetimes [1, 2]. Most die castings are made from non-ferrous metals, such as aluminum, copper, magnesium, lead, zinc, tin-based alloys but also, they can be protected by specific coatings that have high wear resistance. The erosive wear damage in die casting molds is caused by the fact that the molten metal is blown into the mold by high pressure dry air. It is known that the friction between the Al melt and the surface of the casting mold coherently affects mainly the life of the mold and its parts, the quality of the resulting casting and energy consumption [3]. One of the possibilities to increase the service life of the mold for casting Al alloys and its parts is the application of the coating with hard nanocomposite by physical vapor deposition (PVD) coatings based on nitrides. The nitride-based coatings are characterized by high hardness, high melting temperature, good fracture toughness, chemical and physical stability. All coatings, containing transition metal nitride, deposited by PVD, such as AlTiN and AlCrN, have been widely used in industrial applications over the past few decades, especially on cutting tools or molds to prolong the service life and decrease the cost of production. Hard coatings with transition element nitrides have been successfully utilized for applications where specific properties of machining environment are required without compromising the bulk material strengths. In addition to the properties, it is desired to reduce the coefficients of friction (COF) of thin hard coatings under dry conditions for environmental reasons. The tribological problems of machining tools for molding and cutting have triggered the development of protective AlTiN and AlCrN hard

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coatings. To improve the wear resistance and service life, titanium-based and chromium-based nitride coatings has been widely used on cutting tools, molds, and mechanical parts. The AlTiN and AlCrN coatings, developed based on binary TiN and CrN coating, are the typical ternary Ti- and Cr-based coatings [3, 4]. The addition of Al element into the TiN and CrN coatings is beneficial to improve the coating hardness, wear resistance, and high-temperature oxidation resistance. The combination of AlCrN and AlTiN coatings form the quaternary AlTiCrN coatings, combine the advantages of both coatings in abrasive wear resistance and high temperature stability. Multicomponent AlTiCrN coatings synthesized by PVD exhibit improved mechanical performance due to its advanced tribological properties and high temperature oxidation resistance. The oxidation resistance of the AlTiCrN coatings has been reported to be up to 1100°C [4, 5]. The AlCrN based coatings prepared by PVD technology have been widely applied in industrial protection of molds and tools owing to their high hardness (27...32 GPa), high thermal stability (800...900°C) and good tribological behavior [6, 7]. The AlTiCrN is the third-generation coating that is produced by addition of chromium in the AlTiN coating. It is reported that the AlTiCrN coating is superior in surface properties to that of ternary coating. It is due to protective shielding as a result of the formation of stable and dense $\alpha(Al,Cr)_2O_3$ mixed oxides which can withstand the temperature up to 1100°C. In addition, the surface residual stress is suppressed by the formation of CrN sublayer, and this also improves the coating adhesion [8, 9]. The AlTiNs are well known as protective hard coatings with high hardness, good oxidation resistance and high fatigue fracture resistance. They are widely used for protection during the tribological and cutting processes [10, 11].

Material and methods. Böhler K190 Microclean alloy was used for the experiment. The chemical composition of the material (wt%): 2.3 C; 12.5 Cr; 1.1 Mo; 4 V; 0.4 Si; 0.4 Mn. The material is characterized by extremely high wear resistance, excellent toughness, high compressive strength, good stability at high temperatures. The material can be applied in the following areas: pressing and precision cutting; high pressure casting – mold inserts; punching tools; drawing and deep-drawing tools and other. The substrate was prepared by a metallographic process → grinding and polishing. The required substrate roughness was R_a = 0.004 μm. Multilayer coatings AlTiCrN [12] and AlTiN [13] were applied to the base material. The coatings were deposited by PVD by the arc evaporation method on a PLATIT PL1001 Compact device. The device contains 4 planar electrodes, which are in the corner devices. Deposition parameters were as follows: a constant deposition temperature of 430°C was set, the pressure in the vacuum chamber was 0.020 mbar and electric voltage is applied to the electrodes. The bias of the sample was changed to 160 V for the multilayer AlTiN coating and 50 V for the AlTiCrN coating. The chemical composition of multilayer coatings (wt, %): AlTiCrN – 30.6 N; 16.8 Al; 14.15 Ti; 38.45 Cr; AlTiN – 55.5 N; 20.2 Al; 24.3 Ti.

The Pin on ball method was used to evaluate of the COF coatings according to ISO 4600:1992. The test was performed at a temperature of 400°C, under a load of L = 5 N, and speed $v = 4\ cm \cdot s^{-1}$. The path radius r was 2; 4; 8 mm. The ball was made of tungsten carbide (WC) material with a diameter of ∅6 mm, measured COF is calculated as follows

$$\mu = F_t / F_p , \quad (1)$$

where F_t is friction force, N; F_p is normal force, N.

The graphical output of this test is a graph of the dependence of the COF on the path taken by the ball for a specified number of cycles. The wear rate (weight loss) of the coatings is calculated as the difference in weight before and after the test. The volume loss for the ball as well as for the tested coating is also determined.

The relationship is used to determine the loss of the coating volume:

$$V_{disc} = \pi R(S_1 + S_2 + S_3 + S_4) / 2 , \quad (2)$$

where S is impression area, mm^2; R is radius of ball travel on the surface of the test specimen, mm. The following applies to the specific abrasion rate of the coating, $W_{s(disc)}$, ($mm^3 \cdot N^{-1} \cdot m^{-1}$):

$$W_{s(disc)} = V_{disc} / F_p \cdot L, \quad (3)$$

where V_{disc} is wear (loss of volume) of the coating, mm^3; L is total friction distance, m.

Results and discussion. SEM images and thickness of the AlTiCrN and AlTiN coatings are shown in Fig. 1. The SEM fractograph confirms the thickness of the AlTiN (4.119 μm) and the AlTiCrN (4.13 μm) coatings. Both coatings showed dense columnar structure with non-porous and defect-free grain boundaries [14].

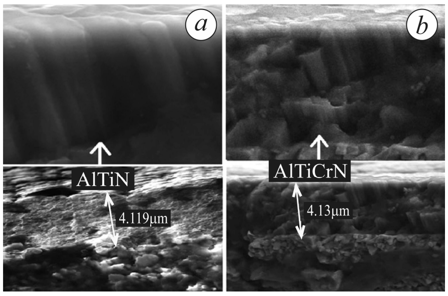

Fig. 1. SEM image of AlTiN (*a*) and AlTiCrN (*b*) coatings [14]. ×20000.

The value of COF increased at the beginning of the test (up to 3000 cycles) and decreased due to the running-in of the friction pair. At the beginning of the test, the microgeometry of the friction pair (coating – ball) was leveled and at the same time the microirregularities on the coating were smoothed at the path of the test ball. After 3000 cycles, the value of the coefficient increased, and after exceeding this value, the COF decreased due to the formation of a path on the sample and a constant value of the load [15, 16]. The lowest value of the COF at 400°C was for a path radius r = 2 mm, where it reached 0.935. For track radius r = 4 and 8 mm, the value of the COF was similar: 0.081 and 0.086, respectively. The dependence of the COF on the track is shown in Fig. 2.

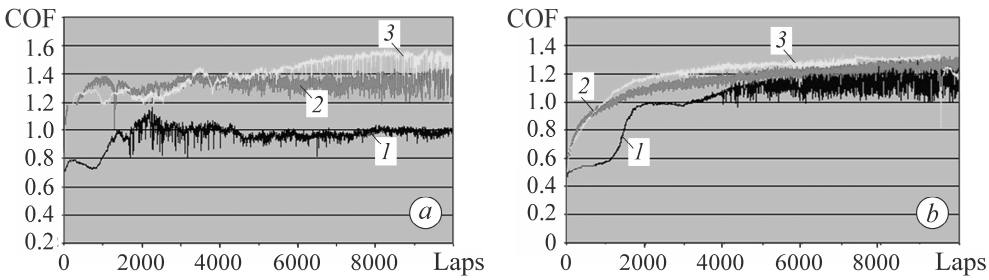

Fig. 2. Dependence of COF vs. sliding distance (Laps) on the AlTiCrN (*a*) and AlTiN (*b*) coatings path: *1* – r = 2 mm; *2* – r = 4 mm; *3* – r = 8 mm.

The values of the COF for the AlTiCrN and AlTiN coatings are shown in Table 1.

Table 1. COF values of AlTiCrN (in numerator) and AlTiN (in denominator) coatings

Track radius r	min	max	Mean	Std.Dev
2 mm	0.75 / 0.438	1.12 / 1.369	0.935 / 1.094	0.081 / 0.148
4 mm	0.558 / 0.448	1.506 / 1.392	1.325 / 1.152	0.086 / 0.132
8 mm	0.576 / 0.447	1.626 / 1.368	1.392 / 1.204	0.126 / 0.144
Average, mm	0.628 / 0.444	1.417 / 1.376	1.217 / 1.150	0.098 / 0.141

The 3D surface topography after tribological test at ×20 magnification of the AlTiCrN and AlTiN coatings is shown in Fig. 3.

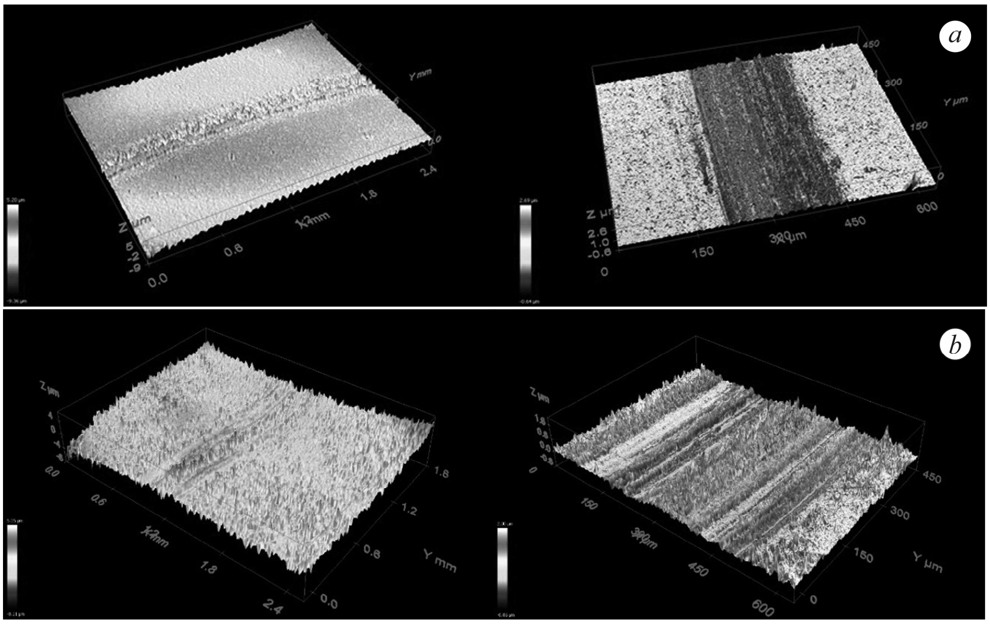

Fig. 3. Surface topography after tribological test of the AlTiCrN (*a*) and AlTiN (*b*) coatings.

At the end of the tribological test, the content of the resulting path and the material deposit, which was formed by sticking the material particles to the ball, were measured [15]. The sites where the AlTiCrN coating material was removed are marked in dark and the places where the particles were deposited on the bead are marked in light. The profilograms of the AlTiCrN and AlTiN coatings are shown in Fig. 4. The values of the material volume removed for the AlTiCrN multilayer coating and the values of the specific abrasion rate *W* at a temperature of 400°C are shown in Table 2.

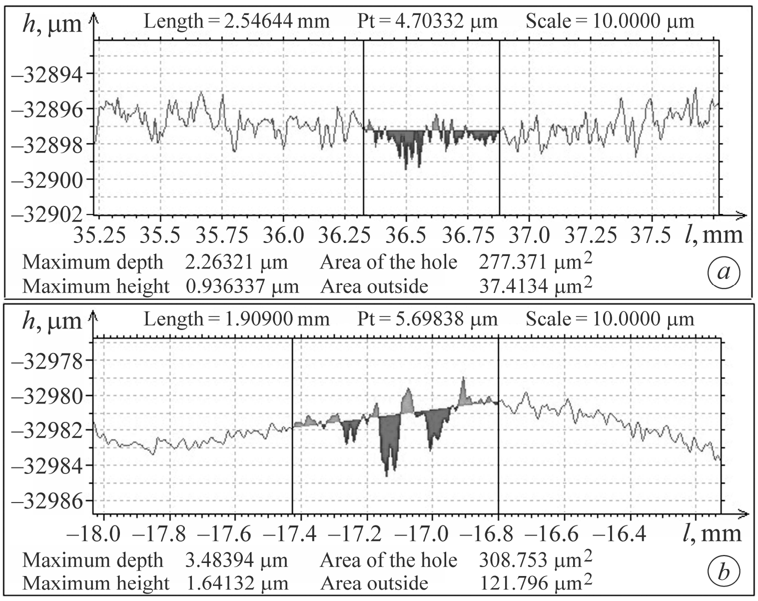

Fig. 4. Profilograms of the AlTiCrN (*a*) and AlTiN (*b*) coatings at the contact point.

In the case of the AlTiN multilayer coating, the lowest COF value 1.094 was recorded for a path radius of 2 mm. The COF on the path is shown in Fig. 3*b*, the radius of the path of the ball did not play an important role in this case. This fact is based on the mean deviation, with the smallest deviation value being measured at r = 2 mm and the largest deviation value being measured at r = 8 mm. From the dependence of the COF on the track, in the initial stages of the test a low value of the COF was achieved due to the running-in of the friction pair. The COF increased to 3000 cycles and after exceeding this value, the course of the COF of the AlTiN coating stabilized and was constant due to the constant load. Compared to the AlTiCrN coating, the AlTiN coating showed better values of the COF [16]. The COF values for the multilayer AlTiN coating are given in Table 1. After tribological test, a profilogram is created, which determines the appearance of the area at the measured site. In the upper part of the profilogram, the profile of the measured area is shown in width, and in the lower part of the profilogram, the surface area of the AlTiN coating is highlighted with the marking of protrusions and depressions [17, 18]. The areas where the material is removed during the Pin on ball test are shown. The profilogram of the AlTiN coating is shown in Fig. 4*b*. The values of the volume of material removed for the AlTiN multilayer coating and the values of the specific abrasion rate *W* at a temperature of 400°C are given in Table 2.

Table 2. Values of the volume of removed material V_{disc} and the specific abrasion rate *W* for the AlTiCrN (in numerator) and AlTiN (in denominator) coatings

AlTiCrN / AlTiN	Ball path radius, mm			Average
	2	4	8	
V_{disc}, mm^3	$\frac{0.00113}{0.00074}$	$\frac{0.00183}{0.00223}$	$\frac{0.00791}{0.00442}$	$\frac{0.00362333}{0.00246300}$
W, mm^3·N^{-1}·m^{-1}	$\frac{1.80 \cdot 10^{-6}}{1.18 \cdot 10^{-6}}$	$\frac{1.5 \cdot 10^{-6}}{1.8 \cdot 10^{-6}}$	$\frac{0.00000315}{0.00000176}$	$\frac{2.137 \cdot 10^{-6}}{1.573 \cdot 10^{-6}}$

CONCLUSION

The COF value of 2 multilayer AlTiCrN and AlTiN coatings was determined and compared. Tribological tests at a temperature of 400°C and at 3 paths of the test ball r = 2; 4; 8 mm were performed. When comparing the COF of both types of coatings, it can be concluded that the multilayer AlTiN coating reached a much lower value compared to the AlTiCrN coating. The size of the radius of the ball travel did not significantly affect the COF value. In the initial phase of the experiments, a low value of the COF was achieved due to the running-in of the friction pair. The value of the COF increased to 3000 cycles and after exceeding this number of cycles, the course of the COF stabilized. After tribological tests, the places where the coating material was removed, respective particles were deposited on the test ball. Even in this case, the AlTiN multilayer coating achieved better values. Two properties were also evaluated and observed: the volume of material removed and the specific abrasion rate. From the performed measurements and the obtained values, it can be concluded that even in this case, better and more favorable values were measured for the multilayer AlTiN coating in comparison with the AlTiCrN coating. From the performed tests and results it can be stated that the AlTiN coating can be used in environments with higher temperatures, i.e. also on the inserts of molds used in high-pressure casting of Al and its alloys.

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