RSM Performance Characteristics Improving Due to the Application of the Nanostructured Carbon-containing Coating "Superlattice"

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Abstract. This article presents the results of the experimental researches on nanostructured carbon-containing PVD coatings based on titanium and chromium (Cr/C; CrN/CNx, Ti/C; TiN/CNx) deposited on a disk Ø 20 x 5 mm made of steel ShKh15 (IIIX15). Coatings were obtained on the UNICOAT 600SL installation (Russia). In the course of the research, such parameters as coatings adhesion to the substrate, their hardness and elastic modulus, as well as friction coefficients were determined by using the indentation, scratch testing and tribological testing methods, respectively, on the CSM equipment (Switzerland). The low values of the friction coefficient (Cr/C – 0,02, Ti/C – 0,07; CrN/CNx - 0,15; TiN/CNx – 0,13) suggest that they are promising to be used in the friction pairs of the drive actuating mechanism, and the high hardness values (from 3,000 to 3,700 units on the HV scale) are promising in highly loaded drives under extreme operating conditions.

1. Introduction
Nanostructured carbon-based superlattice coatings have been of particular interest due to their practical use in friction pairs of actuators and mobile supports [1]. The properties, structure and adhesion [2] of PVD coatings determine a number of factors: degree of ionization; flow velocity and density of the sprayed particles, temperature optimization for coating deposition; application of substrate sputtered cleaning, accelerating stresses, displacements, etc. The presence per se of a solid chemical element (carbon C) in the coating composition cannot be a guarantee of wear resistance improvement. Different deposition modes, tooling configuration, pre-ion etching or alloy addition, and many other features determine the structure of the coatings themselves and the interface boundary structure “coating-substrate” and the coatings properties [3], in contrast to the coatings obtained by laser ablation [4-8, 11, 12].

2. Main part
For comparative experiments, nanostructured carbon-containing coatings [13] (obtained on the UNICOAT 600SL system) based on titanium and chromium [14] (Cr/C; CrN/CNx, Ti/C; TiN/CNx) deposited on a disk (Ø 20 x 5 mm) made of steel ShKh15 (IIIX15) were used. Experimental researches have been carried out on the CSM equipment (Switzerland) using the indentation, scratch testing and tribological testing methods. The main results are given below.

2.1. Obtained coatings
During the superlattice coatings deposition on the UNICOAT 600 SL system, a scheme was used in which two magnetrons are located in the centre of the chamber. With this magnetrons arrangement, the products pass through the treatment zones opposite each magnetron, and an uniform multi-layer periodic structure is created. In the internal part of the chamber there are magnetrons with the atomized target materials Ti and C, (Cr and C) and a table on which the products are located. The table realize planetary movements during the coating deposition.

The engineering process of the superlattice coating deposition consisted of several stages:

2.1.1. Stage 1. Vacuum chamber pumping to the "base" pressure. The purpose of this operation is to obtain a “base” vacuum in the processing chamber. The requirements for the "base" vacuum are determined by two parameters: the pressure magnitude till the $P_{\text{ba}}$ is pumped and the amount of impurity gases and vapours leakage (flow rate) into the vacuum chamber $Q_{\text{n}}$.

2.1.2. Stage 2. Products sputtered cleaning. Products sputtered cleaning is a final cleaning of the products surface in vacuum under the action of surface bombardment with the working gas ions, which are carried out in order to ensure a high coating adhesion. For the sputtered cleaning execution, the working gas (argon) is supplied into the chamber. When the gas pressure is $P_p = 0.20...0.28$ Pa, a glow discharge (the argon ions (Ar$^+$) source) is emitted in the chamber by applying a negative voltage $U_{\text{cm}} = 800...1000$ V to the equipment and by turning on the magnetrons at low discharge currents (up to 1...3 A on every one). This method of sputtered cleaning is called " Magnetron Sputtering Glow Discharge”

2.1.3. Stage 3. Metal precoating deposition. In this operation, a metal layer of titanium (or chromium) is applied, at the values of current $I = 15...20$ A, which ensures a high adhesion between the substrate and the reactive layer

2.1.4. Stage 4. Reactive coating deposition. The vacuum coatings that are formed on the surface of a substrate as a result of a heterogeneous reaction between the deposited metal atoms and the reactive gas molecules (nitrogen, methane, oxygen, etc.) are known as “reactive”. In this operation, internal transient and external reactive layers of coatings are applied using a dual magnetron operating in a bipolar pulsed mode. Reactive layers are applied from two unbalanced magnetrons of the dual system, equipped with specific targets from (Ti, Cr and C), depending on the type of coating obtained. When Cr/C (Ti/C) coatings are obtained, a current $I = 15...20$ A is applied to the magnetrons, and a voltage $u = 40...80$ V to the product. Under these conditions, an intense targets bombardment and an effective transfer of metal and carbon atoms occurs to the work surface, forming a multilayer coating. When CrN/CNx (TiN / CNx) coatings are obtained, a current $I = 15...20$ A, is applied to the magnetrons, and a voltage $u = 40...80$ V, this process occurs under a constant continuous feed of working gas (nitrogen) into the chamber. Under these conditions, an intense targets bombardment, the connection of atoms with a nitrogen gas and the transfer of the obtained nitride compounds to the work surface, forming a multilayer coating.

The cooling products operation is usually carried out at the end of the manufacturing process and is intended to reduce the temperature of the products heated during the coating deposition process prior to the discharge into the atmospheric air chamber. This reduces the influence of some negative factors that can lead to the obtention of defects in the applied coatings.

2.2. Determination of physico-mechanical characteristics

The indentation tests [9] were carried out on the nanoindentometer “CSM Micro scratch tester” using a Vickers diamond indenter. The tests were carried out according to the following method: 1) test specimen fixation on the device; 2) basic setting-up assignment of the indentation mode; 3) indentation execution; 4) photographing of the indentation marking; 5) analysis of the results.

The load application was realized under a certain level (50 mN), then the exposure was carried out for a limited period of time at this load level and unloading (Fig. 1). For each specimen, a series of tests was
performed to determine the following parameters: indenter hardness (Hit), Vickers hardness (HV), given elastic modulus (E*).

![Indentation curves](image)

**Figure. 1.** Indentation curves under at a maximum load 50 mN

The software allowed to automatically process the results and build the “load-penetration depth” diagrams using the Oliver-Pharr method. The quantitative estimation of the examined coatings was carried out on the basis of these curves (Fig. 1) and indentation impress. Fig. 2 shows an example of an impress with a size of 3 x 3 μm; Table 1 shows the average values of the physico-mechanical characteristics for the examined specimens.

![Impress indentation marking](image)

**Figure. 2.** Impress indentation marking (x50)

**Table 1.** Indentation measurement results

| Coating     | Hit, GPa | E*, GPa | HV    |
|-------------|----------|---------|-------|
| TiN/CNx     | 39       | 250     | 3700  |
| Ti/C        | 35       | 280     | 3450  |
| Cr N/CNx    | 31       | 310     | 2970  |
| Cr/C        | 39       | 230     | 3690  |
The scratch method testing was carried out on the nanoindentometer “CSM Micro scratch tester” using a Rockwell diamond indenter and linear scratching. According to the results, it is concluded the critical load at which the coating begins to split off from the substrate.

The tests were carried out according to the following method: 1) fixing the test sample on the device; 2) preliminary scanning of the surface and determination of its profile; 3) parameters assignment for scratch testing in the settings; 4) scratch testing execution; 5) panoramic image building of the scratch marking; 6) analysis of the results.

The software allowed in automatic mode to process the obtained diagrams “load penetration depth - acoustic emission signal”. The scratch testing parameters were the same for all specimens: initial load 0,1 N, final load 15 N; load rate 8,94 N/min; scanning load 0,03 N; scanning load speed 2 mm/min; measurement length 5 mm; indenter radius 100 μm. Fig. 3 presents the results of scratch testing. The numerical values of the scratch tests are shown in Table. 2.

Table 2. Scratch testing and tribometrics results

| Test № | Load at which the first shearing distortion is formed, H | Friction coefficient value |
|--------|------------------------------------------------------|----------------------------|
| Cr/C   | 4,64                                                 | 0,025                      |
| CrN/CNx| 4,85                                                 | 0,15                       |
| Ti/C   | 5,83                                                 | 0,072                      |
| CrN/CNx| 5,69                                                 | 0,13                       |

Tribological tests were carried out according to the following procedure: 1) fixing the test specimen on the swivel slide; 2) adjustment of the movable rod and fixing on it a rotating ball-indenter; 3) setting testing parameters (material, specimen speed, ball pressing force, ambient temperature, test time, etc.); 4) tests execution; 5) loads graphs obtaintion, abrasion depth and friction coefficient; 6) wear track research and analysis of the obtained results.

The tests of all three coatings were carried out with the following identical parameters: track diameter 11 mm, load on the specimen 10 N, sample speed 20 cm/s, travel 300 m, the counterbody material was steel ShKh15 (ШК15), the counterbody geometry was a ball with a diameter of 6 mm. Fig. 4 presents the friction coefficient changes graphs for the investigated coatings and the average values of the friction coefficient are shown in Table 2.
Fig. 3. Scratch testing results
3. Conclusions

The analysis of the experimental results shows that the hardness of the investigated coatings ranges from 3,000 to 3,700 units on the HV scale, and it is not inferior than the known coatings, even than those which do not contain carbon (for example, the traditional coatings TiN 2900 HV, TiAlN 3200 HV).

The modulus of elasticity of the investigated coatings shows low values: 230 - 310 GPa (in comparison: TiN 361 GPa, TiAlN 650 GPa).

The realized tribological tests revealed low friction coefficient values for coatings based on C. For the coating (AlSiCr)C: the coefficient of friction Cr/C is 0,02, Ti/C is 0,07, CrN/CNx is 0,15, TiN/CNx is 0,13 (in comparison, the TiN and TiAlN coefficients are much larger).

Low friction coefficient values allow to suggest to use carbon-containing coatings in moving friction pairs and actuating mechanisms. High values of hardness characteristics allow to use these coatings in high-loaded drives and extreme operating conditions.

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