Nanostructured wear-resistant coating for the composite turning inserts

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Abstract. The article presents the results of study of microstructural and strength properties of developed nanostructured wear-resistant high-hardness coating with high strength characteristics, resistance to shock and vibration loads on the surface of cutting inserts for instrumental and high-speed steel turning treatment. The specificity of the turning operating conditions of the composite cutting tool inserts shows that the cutting inserts working surface should have such characteristics as high hardness, strengths, increased vibration resistance, resistance dynamic loads and thermal shock, as well as increased wear resistance. Therefore, one of the ways to solve this problem proposed in the report is applying a wear-resistant hard oxide phase nanostructured coating on the surface of the cutting edge of the composite cutting inserts. Technological modes were selected for the wear-resistant hard protective coating which provides high microhardness due to the nanostructure by magnetron sputtering deposition of the developed compositions that have high adhesion to substrates made of composite cutting turning inserts. The results are shown on the example of high-speed turning of 30KhGSA by using composite turning inserts with developed nanostructured wear-resistant hard protective coating.

1. Introduction

It is known that thermal load on cutting tool has a great influence on the tool wear, tool life and machined surface quality [1, 2]. It was also stated that all the cutting heat generated in the cutting zone during dry cutting manipulation is almost conducted into chips, tool and workpiece [1]. There are several important cutting process issues such as cutting speed regime, presence of coolant fluid, cutting temperature growth, cutting force regime, cutting tool life characteristics and machined surface integrity are great influenced on heat flow into cutting tool [3, 4].

The increase of degree of the heat partition into cutting tool gives the significant cutting tool temperature elevation that accelerates cutting tool chip wear and degrades the efficiency of turning metal materials [2]. As it is known, the heat transfer into cutting tool is managed by many factors such as cutting fluid feeding mode, main cutting parameters, workpiece and cutting tool thermo-physical properties [2]. The technique of use of cutting fluids in metal cutting-turning process decreased the cutting tool temperature by reducing the generation of thermomechanical energy as well as convection cooling [5]. However the cutting fluids are reduced or avoided in series of metal-turning process and...
The dry cutting method is widely used nowadays. The percentage of cutting heat energy entering chips, workpiece and cutting tool varies significantly; and according to Fleischer [6] the percentage of cutting heat flowing into the cutting tool varies from 2% to 18%, into the workpiece from 1% to 20% and into the chips from 74% to 96%.

The previous evaluation [7] of the thermomechanical energy partition in the process of orthogonal dry turning of AISI 1045 carbon steel and AISI 304 austenitic stainless steel with three different cutting tools (CVD-TiC/Al₂O₃/TiN, TiC/TiCN/Al₂O₃/TiN and uncoated carbide cutting tools) was showed that workpiece and cutting tool material surface properties greatly affected the heat partition. The high-hard coated cutting tools presented smaller heat energy partition into cutting tool compared with the uncoated carbide cutting tool [7].

Thus, the development of nanocomposite from the highly hard and refractory phases such as Al₂O₃, Cr₂O₃, TiN, TiC, TiB₂ is one of the recognized directions for solving the problem of thermomechanical wear of metalworking turning tools [8, 9]. This paper discusses the results of development for nanostructured high-hard refractory wear-resistant protective coating on their surface of composite turning inserts by the method of magnetron- plasma sputtering, which allows changing significantly the physical and mechanical characteristics of the material for the cutting edge of cutter inserts. Coating by ion-magnetron sputtering, which allows spraying thin nanostructured films, is currently the most optimal low-cost technology for obtaining wear-resistant protective coatings on commercial cutting tools and replaceable turning inserts, both in terms of adhesion and the resulting structure of the sprayed nanostructured coating.

2. Materials and equipment
The following equipment was used for research: commercially available composite replaceable cutting lathe inserts (China). A Falcon 500 microhardness tester was used for microhardness tests during a preliminary wear resistance analysis. A Q150T ES magnetron plasma spraying unit was used to apply nanostructured coatings. An industrial unit by VOSOCO Ozone Generator (China) was used for ozone phase-forming annealing of metal / alloy films. Video microscope Supereyes T004 250X-2000X USB 5.0 MP was used to study the surface and structure of composite turning plates. A scanning electron microscope JEOL JSM-7500F and a Perkin-Elmer Spectrum Two FTIR spectrometer were used to study the microstructure of the coatings and analyze the composition of the coatings.

3. Analysis of experimental data
The optical images of the side and front part of a composite cutting tool replaceable turning inserts obtained with a white light source and UV-range (380 nm) source are shown in Figure 1.

![Figure 1. The optical images of the side and nose part composite turning insert illuminated by white and UV light source.](image-url)

The intermediate part of the used commercially available turning insert with a black sintered material is a micropowder of boron carbide and aluminum oxide in a smaller proportion bonded a copper-aluminum bond. The center and outer part of the cutter with a gap of abrasive material consists of HSS steel.

The microstructure of the surface of different parts of composite turning insert was studied using scanning electron microscope and shown in Figure 2.
Figure 2. The SEM images of the surface of left to right central part, abrasive layer part and side part composite turning insert

To apply nanostructured wear-resistant high-hard protective coatings by magnetron sputtering on the surface of composite turning insert the next technological cycle is used: 1) preliminary etching of the composite turning insert surface with low-temperature argon plasma to improve the adhesion of the applied protective coating; 2) sputtering by the method of magnetron-plasma spraying of a nanostructured film of metal or alloy on the surfaces of the turning insert; 3) thermal oxidative ozone-assisted phase-forming annealing for the appearance of nanoparticles of high-hard oxides of a cubic system (aluminum or chromium oxides) and nanoparticles of medium-hard oxides (oxides of titanium and vanadium), which also leads to an increase in the adhesion of the protective coating, an increase in hardness, wear resistance and fatigue strength due to the recrystallization process applied coating.

Preliminary treatment of the surface of the composite turning insert with low-temperature argon plasma makes it easy to clean the surface of the material substrate from dirt and grease residues. Also argon ion-plasma etching makes change the structural and mechanical properties of the material substrate, increase the roughness, which will improve the adhesion between the metal surface of the turning insert and the applied sputtered material.

With the help of magnetron-plasma spraying unit the nanostructured metal thin-layer films of the desired chemical composition and thickness are deposited in vertical direction upright horizontal surface of composite turning insert. For the deposition of nanostructured metal coatings 100 nm thick on the surface of the cutting edge of high-speed steel tools, a Q150T ES RF magnetron sputtering unit with appropriate metal targets was used. The following values of magnetron sputtering currents were used at weak discharge (2×10⁻⁴ Pa): 50 mA for chromium, 120 mA for Ti6Al4V alloy, 120 mA for aluminum. With the described method of obtaining the preliminary coating, no thermal heating of the turning insert observed, due to which there is no occurrence of residual stresses on the surface of the cutting edge and along the product-coating interface.

Usually radio frequency-driven magnetron-plasma spraying occurring at room temperature without substrate heating provides a metal coating having a nanoscale structure in the size range of 8-15 nm [8]. Thin nanostructured coatings, with a certain crystal structure (cubic and hexagonal syngonies) and size of nanoparticles belonging to the region of maximum realization of the Hall-Petch effect show ultra-high hardness, high fatigue strength, and increased wear resistance.

The microstructure of the obtained metal/alloy coatings on the surface of composite turning insert was studied using scanning electron microscope and shown in Figure 3.

Figure 3. Microstructure of the obtained coatings: a - chrome; b - Ti6Al4V alloy; c - thermally oxidized aluminum (Al₂O₃) (left image - before thermal annealing; right image - after oxidative thermal annealing).
Thermal oxidative phase-forming annealing makes it possible to create a surface layer of highly hard, highly thermally stable cubic or tetragonal phase oxide nanoparticles on the surface of a metal adhesive film, which leads to the production of highly hard coatings with high wear resistance and significant fatigue strength. The low temperature ozone-assisted thermal oxidative phase-forming annealing is carried out at a temperature of 350 °C for 1.5 hours for the appearance in coatings of nanoparticles of high-hard oxides of a cubic or tetragonal system (oxides of aluminum, chromium, titanium, etc.), which also leads to an increase in the adhesion of the protective coating, an increase in hardness and wear resistance due to the process of recrystallization of the applied coating. As a proof of concept, studies of the hardness of produced nanostructured coatings with nanoparticles of organized high-strength oxide films of cubic or tetragonal phases on the surface of high-speed steel turning insert were carried out.

The nanosized aluminum, chromium and Ti6Al4V alloy film was annealed at condition of ozone-assisted thermal oxidative at temperature of 350 °C to converted to the oxide phase according to FTIR spectroscopy data. The microstructure of the resulted oxide coatings was studied using scanning electron microscope and shown in Figure 3.

According to electron microscopy data, the nanostructured coating of ozone-assisted thermally oxidized chromium exhibits the highest micro-roughness about value $S_d = 3.9$ nm. The nanostructured coating of ozone-assisted thermally oxidized Ti6Al4V alloy exhibits the micro-roughness about value $S_d = 3.3$ nm. Also, the nanostructured coating of ozone-assisted thermally oxidized aluminum ($Al_2O_3$) exhibits the modest micro-roughness about value $S_d = 2.5$ nm. It can be seen that the fabricated oxide coatings contain clusters of nanoparticles 20–40 nm in size and the size of clusters of nanoparticles in different coatings is different. The calculated with Gwyddion 2.54 program values of mean size of nanoparticles and fractal dimension in cube counting approximation for derived coatings is shown coatings are presented in Table 1.

**Table 1.** Average size of nanoparticles and fractal dimension in nanostructured coatings.

| properties       | chromium | $Cr_2O_3$ | $Al_2O_3$ | Ti6Al4V | (Ti6Al4V)Ox |
|------------------|----------|-----------|-----------|---------|-------------|
| mean size, nm    | 10±3     | 23±5      | 28±6      | 14±4    | 24±5        |
| fractal dimension| 2.59     | 2.57      | 2.56      | 2.60    | 2.53        |

The observed decrease of fractal dimension for produced nanostructured oxide films occurs due to the oxidation process with particle recrystallization. The decrease of fractal dimension of thermal oxidation annealed films can be interpreted as the appearance of nanoparticles with flatter faces.

The derived nanostructured $Cr_2O_3$ and $Al_2O_3$ coating shows a wide-range distribution of nanoparticles in size, which is characteristic of secondary crystallization processes due to the oxidation of metal nanoparticles with remelting and coalescence [10]. It can be assumed that, in accordance with the well-known Hall-Petch equation [11], the size of the nanoparticles of the deposited alloy films and the resulting chromia and alumina nanoparticles is close to the transition boundary from the forward Hall-Petch law to the reverse one [12]. Thus, a high microhardness can be expected for the obtained nanostructured refractory oxide coatings. The Rockwell microhardness of the obtained coating metal and oxide samples was measured and microhardness values were taken as averages over the results of 6 measurements (Table 2).

**Table 2.** Rockwell microhardness of samples of the initial HSS material of composite cutting turning insert and with applied nanostructured coatings.

| turning insert | chromium | $Cr_2O_3$ | $Al_2O_3$ | Ti6Al4V | (Ti6Al4V)Ox |
|----------------|----------|-----------|-----------|---------|-------------|
| HSS            | 57±2     | 58±3      | 64±2      | 70±2    | 60±2        | 67±2        |

From the data in Table 2, it follows that, with the exception of the chromium film, the nanostructured coatings of metals and derived oxides noticeably increase the hardness of the working surfaces of the HSS surface of composite turning insert. For the case of a nanostructured film coating of $Al_2O_3$ obtained by thermal oxidation of aluminum, high hardness is quite expected for hexagonal α-
phase or cubic phase γ-Al₂O₃. In the case of the prepared coating of the Ti6Al4V alloy and their mixed oxide film, the obtained considerable microhardness result is rather unexpected. Nevertheless, according to the data of studies, a decrease in the grain size in the Ti6Al4V alloy [13] leads to a strong increase in its strength and plasticity. Thus, we can concluded that after deposition of metal film the following ozone-assisted thermal oxidative phase-forming annealing of Ti6Al4V alloy film lead to the appearance of highly hard γ-Al₂O₃ nanoparticles of cubic syngony with admixtures of the phases of oxide nanoparticles TiO₂ and VO₃, which also leads to an increase in the adhesion of the protective coating, an increase in hardness and wear resistance due to the process of recrystallization of the applied coating.

In according to SEM microscopy results the pronounced change in the microstructure of magnetron-sputtered protective cermet coatings occurs both from the oxidation of metal nanoparticles according to IR spectroscopy data and due to recrystallization of the formed oxide phases. In all cases studied by us, the obtained samples of oxide protective coatings are nanostructured with a nanoparticle size from 15 to 35 nm.

Thus, the preliminary results show the validity of the proposed approach, since the developed single-layer nanostructured coatings noticeably increase the hardness of the working surfaces of high-speed steel composite turning insert.

It is known that the main crystalline phases of TiO₂ are tetragonal anatase and rutile phases. At low-temperature oxidation up to 400°C, the anatase phase predominates, and at temperatures above 600°C anatase is almost completely transformed into a thermodynamically more stable and harder rutile phase. Based on the thermal annealing conditions we used, it can be assumed that the basis of the created nanostructured wear-resistant high-hard protective coatings is a film of medium-hard rutile nanoparticles interspersed with high-hard nanoparticles of the cubic phase γ-Al₂O₃, the pseudohexagonal medium-hard phase of VO₂.

The results of testing the manufactured nanostructured wear-resistant protective coatings on composite HSS-based turning insert made with B₄C and Al₂O₃ abrasive micropowders cemented with Cu-Al binder by the example of dry turning of cylindrical parts made of structural alloy steel 30HGSA are shown in Table 3. The tests were carried out for a series of 5 pieces of turning insert with applied wear-resistant protective coatings until the cutter plates failed to obtain provide the required level of surface roughness quality of cylindrical workpiece. The service life of developed composite turning insert with protective nanostructured coatings during lathe turning process was estimated by averaging over five time measurements.

Analysis of the data presented in the table 3 allows us to conclude that cutting tool composite chip with nanostructured wear-resistant coatings are characterized by higher physical and mechanical characteristics and a longer service life (up to 23% of the time when machine turning of steel 30HGSA workpiece), compared with original uncoated metal-ceramic composite turning insert.

**Table 3.** Results of testing the service life of composite cutting turning inserts with manufactured protective wear-resistant coatings.

| Sample coating | Treatment resistance for 30KhGSA, min. |
|----------------|----------------------------------------|
| uncoated       | 56±2                                   |
| Cr₂O₃          | 62±2                                   |
| Al₂O₃          | 67±3                                   |
| (Ti6Al4V)O₄    | 69±3                                   |

The reasons for difference of longer service life enhancement for tested protective wear-resistant coating this may be the complex cooperative nature of interparticle and interphase interactions in the created protective film coatings at thermo-mechanical wear. Thus, for example, upon oxidative phase-forming annealing in air-ozone flow in nanostructured Ti6Al4V films, in addition to nanoparticles of various Al₂O₃ phases, the nanoparticles of hard phases of titania and vanadium oxides with various combinations of hardness and plasticity can be formed.
4. Conclusion
As a result of the research, samples of high-hard refractory wear-resistant nanostructured coating for cutting tool of composite turning insert developed. Obtaining a protective coating on the surface of the turning insert is carried out by the method of magnetron-plasma sputtering, including preliminary plasma etching of the surface of the cutting tool in a vacuum chamber with accelerated argon ions. The followed phase-forming ozone-assisting thermal oxidative annealing to derived refractory hard wear-resistant coating of high-hard nanoparticles of Al2O3, Cr2O3, titanium/aluminium/vanadium oxides.

5. References
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