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## Improvement of the Physical and Mechanical Properties of the Cutting Tool by Applying Wear-resistant Coatings Based on Ti, Al, Si, and N

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**Abstract.** From the great variety of methods to improve the efficiency of cutting tools, it is necessary to highlight the methods of applying wear-resistant coatings, which in recent years are increasingly used. Applying wear-resistant coatings on the cutting tool can significantly increase its efficiency and intensify machining modes. Mechanisms of strengthening the wear-resistant coating for materials have been analyzed under the impact of technological parameters of coating condensation process on its structure parameters and mechanical properties, formation of single and multi-element coatings based on titanium nitrides, aluminum, and silicon, the transformation of coating properties by obtaining complex coatings, and principles formation of complex coatings designed for different cutting tools. The influence of the coating on the mechanical properties of high-speed steel is shown. In the magnetron sputtering coatings on P6M5 steel samples, the microhardness of the coatings is TiN – 20–24 GPa, AlN – up to 16 GPa, TiAlN – up to 35 GPa, AlTiN – up to 32 GPa, TiAlSiN – 32–37 GPa, including while the microhardness of the substrate of steel P6M5 – 6–9 GPa. The microhardness of TiAlN and TiAlSiN coatings applied on an instrumental basis is 1,5 - 1,9 times higher than the microhardness of TiN, AlN coatings. It was found that the wear intensity of P6M5 steel without coating is 6 times higher than with AlTiN, TiAlN, and TiAlSiN coating, 3 times higher than with TiN and AlN coating. The coated tool is characterized by increased reliability and higher stability and allows the processing process with higher cutting modes.

**Keywords:** coating, cutting tool, structure, properties, wear resistance.

## 1 Introduction

The low life of tools and machine parts is mainly due to rapid wear and corrosion damage to their surface layers.

To eliminate or inhibit the processes that adversely affect the performance of products, use various methods of targeted changes in the properties of the surface layers of materials. One of the effective ways to increase the efficiency of the cutting tool is to apply wear-resistant coatings to its working surfaces. Wear-resistant coatings allow the tool to obtain working surfaces with the required performance characteristics, usually without changing the properties of the base material.

Advantages of application of the cutting tool with a covering: transition to higher modes of cutting; increase the service life of the tool; improving the quality of the treated surface; efficient use of equipment; reducing the cost of products [1].

Based on the operating conditions of the tools, wear-resistant coatings must have: high hardness and store it at cutting temperatures; inertness to adhesion and high-temperature corrosion with the processed material in the whole range of cutting temperatures; stability of mechanical properties up to the temperature of heat resistance of tool material; inertness to dissolution in the processed material at high temperatures; resistance to failure at significant fluctuations in temperature and stress [2-4].

The urgency of the work is to study the effect of nanostructured wear-resistant coatings based on nitrides Ti, Al, Si, obtained by reactive magnetron sputtering, on the structural-phase state, mechanical and tribological properties of materials for cutting tools.

Wear-resistant coatings, having a lower coefficient of friction than the tool substrate and a higher stability temperature, significantly affect the various parameters of the cutting process. In particular, reduce the length of elastic and plastic contact of chips with the tool's

working surfaces, reduce the cutting force, reduce the temperature in the machining area, change the angle of the conditional shear plane, and, consequently, affect the formation of surface layer quality.

In this regard, the development of the coating composition, which has high strength characteristics, wear-resistant and tribological properties, is an urgent scientific and technical task.

## 2 Literature Review

In modern production, wear-resistant coatings of cutting tools are subject to increased requirements due to complex factors. These factors are related to improving the properties of coatings, the composition of the “coating – tool material”, and the optimization of the operating conditions of the cutting tool. Optimization of conditions of use of the tool relates to a decrease in the growth and intensity of adhesive-fatigue processes, increase in rigidity of the design of the tool and the machine tool equipment. Coatings can be considered an intermediate technological environment between tools and processed materials [3, 4].

Based on this provision, the general requirements for protective coatings have been systematized, which are the basis of some specific coating requirements for cutting tools. Such requirements can be classified into three main groups based on the provision that coverage should [4]:

- overtime to function stably on the contact pads of the tool without premature failure (the role of the coating as a single solid body);
- to provide favorable transformation of contact processes at cutting (official purpose of a covering);
- have a strong adhesive bond with the substrate material (substrate) (tool materials and coating materials must function as a single solid body).

Refractory metal-metalloid (Me-X) compounds are used as wear-resistant coating materials, where refractory metals of groups IV-VI of the Periodic Table of the Elements are used as a metal, and carbon, nitrogen, and oxygen are used as metalloids.

There is a three-level division of factors that determine the properties of coatings [3-6]:

1. Factors related to the coating process parameters (e.g., substrate temperature and the process itself, the interaction of reagents and reaction products, mechanical stresses arising in the coating due to thermal and shock action by a bombardment of streams, and various particles).

2. Factors related to the condition of the substrate, the coating, and the entire system “substrate – coating” in general.

3. Factors associated with structural strengthening (microstructure), including grain size and orientation, grain boundaries, density (porosity).

ZrN, CrN, TiN, AlN, BN, Mo(W)S<sub>2</sub> coatings were widely researched. Those coatings and modified TiAlN, AlCrN, and other coatings and successfully used in industry [3, 7].

The following areas of improvement of the cutting tool with wear-resistant coatings are considered [5, 6, 8, 9]: improved coating technology and new coating compositions, development, and improved multilayer coating structures.

Thus applied nitride coatings of TiAlN, TiCN, TiZrCN, TiZrN increase the period of stability of end mills with carbide inserts MK8 in 1.5–3.0 times in comparison with the cutting tool with TiN coating in the processing of billets of steel 5HNM, 18H18N10T and titanium alloy [8, 10-11].

The influence of the parameters of coating TiN and ZrN by methods of magnetron sputtering and CIB on their hardness is considered in the works [2, 8, 10, 12-16]. The maximum hardness of coatings at a stoichiometry of structure reached by optimization of technological parameters – the pressure of reaction gas, bias voltage, induction of a magnetic field, and discharge current is noted.

The influence of condensation temperature in the coating process is considered in the works [15-18]. A method of increasing the efficiency of the cutting tool by applying coatings in the combined temperature mode is proposed. The temperature of the coating affects its defect and, consequently, its strength and hardness, as well as the strength of the adhesive bond with the tool base and the level of residual stresses. Deposition of the lower layer of the coating at high process temperatures helps to increase the strength of adhesion to the tool base and the formation of the upper layer at lower temperatures, increasing the hardness and wear resistance of the coating.

The authors of the work [19] consider the effect of plasma flow separation on the structure, physical and mechanical characteristics, and wear resistance of TiN coating. Reducing the content of the dripping phase, improving the structural characteristics lead to an increase in wear resistance of such coatings by 1.5–2.0 times.

Coatings based on modified titanium nitride have become the most widespread in world practice. Zirconium, aluminum, molybdenum, chromium, iron, and silicon are alloying elements. [3, 6, 20]. The influence of alloying elements is manifested in the improvement of the physical and mechanical properties of coatings, changing the conditions of contact interaction of the coating with the processed material and the strength of its adhesion to the tool base. Studies of physicomechanical properties of TiN coatings doped with molybdenum, chromium, and silicon [3, 6, 20], have shown that doping with these elements increases the microhardness of coatings by 18-25 %, residual compressive stresses – 1.3–1.9 times; at the same time, the strength of adhesion to the tool base decreases, as evidenced by the increase in the coefficient of exfoliation by 35-80 %.

The study of the influence of the composition of TiMoN coating obtained from separate cathodes on the physical and mechanical properties and performance of the coated cutting tool was carried out in the following work [20]. The studied coatings have a 1.5 to 1.6 times

higher microhardness than TiN coatings. There is a decrease in the bond strength with the tool material for the coating based on titanium nitride and molybdenum. The use of TiMoN coating allowed increasing the period of stability of carbide plates when turning workpieces from steels 30HGSA and 12H18N10T in 1.2–2.2 times in comparison with one-element TiN coating. The use of silicon as an alloying element has a number of advantages [20].

Therefore, this work aims to establish the influence of physical and technological parameters of deposition, working gas pressure, and shear potential on the formation of nanostructured nitride coatings Ti, Al, Si, and N, establishing their structural-phase state, mechanical and tribological characteristics, as in the use of single-element coatings, and as in the use of complex coatings.

It is necessary to determine the nature of the material's behavior in operating conditions before and after coating.

### 3 Research Methodology

#### 3.1 Research objects

The cutting tool (Figure 1) on which the coating is applied has types that conditionally classify it as manual and machine.



Figure 1 – Varieties of cutting tools [21]

Currently used tool materials are divided into the following groups: carbon and low-alloy tool steels, fast-cutting steels, hard alloys, mineral ceramics and cermets, synthetic compositions of boron nitride, synthetic and natural diamonds.

Depending on the materials for the cutting tool, the coating methods shown in Figure 2 are used [22].

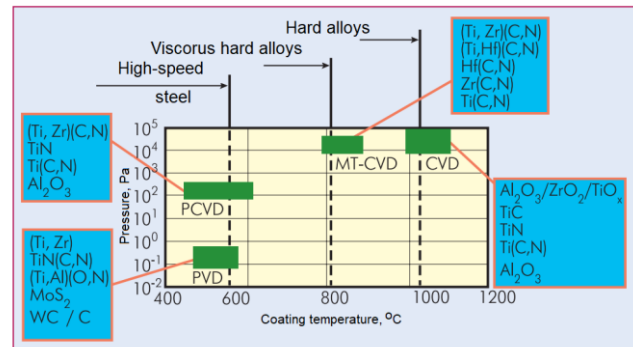


Figure 2 – Parameters and basic methods of coating [22]

Within these groups, there are a large number of coating methods, including combined or methods with support or activation of the process from other energy sources.

#### 3.2 Research methods

A variety of PVD methods based on glow discharge is magnetron sputtering. Magnetron ion sputtering systems are diode-type sputtering systems in which atoms of the sprayed material are removed from the surface of the target by bombarding it with ions of working gas (usually argon) formed in the plasma of an abnormal glow discharge. For increasing the spray rate, it is necessary to increase the intensity of ion bombardment of the target, i.e., the ionic current density on the surface of the target [2, 8].

Magnetron sputtering (DCMS) refers to sputtering materials by ion bombardment.

The main elements of the magnetron sputtering system are a flat cathode made of sprayed material, an anode mounted on the perimeter of the cathode, a magnetic system (usually based on permanent magnets), and a water-cooling system (Figure 3).

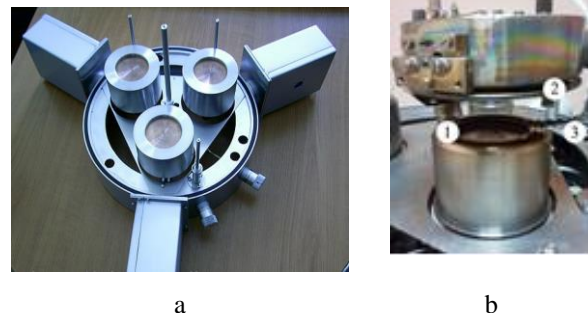


Figure 3 – Magnetron for vacuum deposition of coatings: a – appearance; b – an arrangement in GDP-5M; 1 – one of the targets; 2 – holder for samples with the possibility of rotation; 3 – is the gas supply pipe between the sample holder and the magnetron [2, 8]



Magnetron sputtering of titanium, aluminum, and silicon in the reaction medium of nitrogen and argon allows obtaining coatings that do not contain hydrogen, oxide, and other impurities.

Gases were used for the processes of magnetron sputtering of coatings:

- gaseous nitrogen of exceptional purity 99.999 % (GOST 9293-04);
- argon gaseous high purity of 99.999% (GOST 10157-07).

The uniformity of the composition in the thickness of the coating will be high, and at the lowest thicknesses, the porosity will be minimal. The condensation rate varied depending on the voltage applied to the target and the gas flow.

Investigations of the structure and properties of TiN, AlN, TiAlN, and TiAlSiN films deposited by magnetron sputtering at the VUP-5M unit at a residual pressure in the working chamber  $10^{-3} - 10^{-4}$  Pa were performed on samples of fast-cutting steel P6M5 after standard heat treatment and polished plates of monocrystalline silicon. The rate of deposition and measurement of the thickness of the coatings was controlled by the in-situ method using a quartz thickness gauge according to standard methods.

P6M5 steel substrates were placed over the magnetron with subsequent table rotation to ensure uniformity of coatings in thickness. Before the application process, the vacuum chamber was pumped to a residual pressure of  $1 \cdot 10^{-3}$  Pa. The formation of a gas mixture of argon and nitrogen in the required proportions took place from independent sources in the spraying process. For removing uncontrolled contamination of the target surface and substrates, short-term etching with argon bombardment ions was used.

The coatings' structure was studied using a Tescan VEGA 3 scanning electron microscope (EDX spectra were obtained using additional detectors on the same microscope), a PEM 125K transmission electron microscope, and atomic force microscopy (AFM).

Tests for wear resistance of coatings were carried out under conditions of "dry" friction on a friction machine SMT-1, which is designed for testing for friction and wear, to study the friction and wear processes of metals, alloys, and rigid structural plastics. Counter-body material - hard alloy coated with cubic boron nitride, hardness of counter-body – 80 HRC, dimensions of counter-la – Ø 30 mm, load on the sample (F) – 2 941 g/cm<sup>2</sup>, friction path of test – 2 km, number of revolutions of the counterbody – 355 rpm, test time – 60 and 120 minutes.

### 3.3 Heat treatment of P6M5 steel substrate for applying coatings

Research of the structure and properties of TiN, AlN, TiAlN, and TiAlSiN coatings deposited by magnetron sputtering at the VUP-5M unit were performed on P6M5 high-speed steel samples after standard heat treatment.

The chemical composition of the substrate P6M5 is shown in Table 1.

Table 1 – Chemical composition of steel P6M5, %

C	Si	Mn	Ni	S	P	Cr	Mo	W	V	Co
0.82	0.30	0.44	0.35	0.008	0.027	4.34	4.83	5.91	1.94	0.27

Standard heat treatment of P6M5 steel consists of preliminary heat treatment – isothermal annealing for 6 hours; final heat treatment – hardening with two heats for 0.75 hours and three times for 1 hour each (Figure 4).

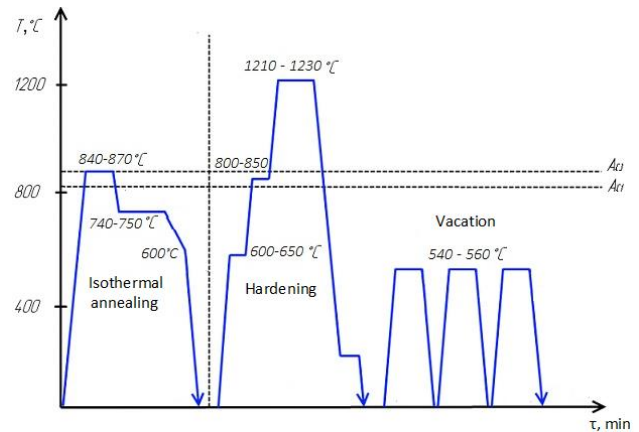


Figure 4 - Graph of heat treatment of experimental plates made of P6M5 steel

After proper heat treatment, the steel had a martensite structure with evenly distributed small and medium-sized carbides (Figure 5).

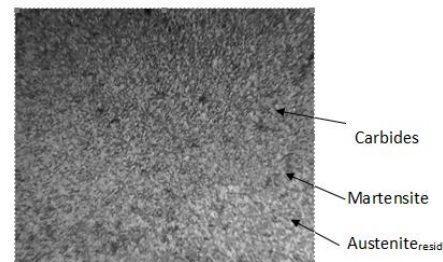


Figure 5 – Microstructure of P6M5 steel after heat treatment (isothermal annealing, quenching with heating, triple tempering), x500

The hardness of the tool after tempering was 63–65 HRC. The proposed final heat treatment reduces the amount of residual austenite in the steel and increases the wear resistance of the tool by 2.5–4.0 times.

Before deposition of the coatings, the substrates were polished by a mechanical method combined with a diamond grinding wheel. Then the substrates were sonicated for 10 minutes, successively in acetone and ethyl alcohol.

## 4 Results

### 4.1 Study of coatings

#### 4.1.1 Study of TiN coatings

Titanium nitride TiN is a widely used, traditional compound applied to carbon, stainless and fast-cutting steels, and hard alloys and ceramics. In metalworking, the tool's temperature is reduced due to the high surface

density of titanium nitride. TiN was one of the first-facing materials used in industrial cutting tools, and it is still used as diffusion barriers and for decorative coatings.

The material can be obtained by physical vapor deposition (PVD) and chemicals. Like most other ceramic materials, it has a brilliant appearance and golden color, relatively good mechanical and thermal properties. Titanium nitride is oxidized at a reasonably high temperature (above 450 °C), one of its main disadvantages when used as a coating tool. Evaporation of the nitride coating in an inert atmosphere leads to a decrease in its internal hardness due to the annihilation of defects and stress relaxation.

To study the effect of deposition parameters on the characteristics of titanium nitride coatings applied to substrates - polished plates of monocrystalline silicon Si using a magnetron spray system on a universal vacuum unit GDP 5M in Ar and N<sub>2</sub>, changing the flow rate of nitrogen into the working chamber and the distance between target and substrate “L<sub>s-c</sub>” (s – substrate, c – coating). The deposition was carried out in the mode of power limitation (3 kW) while maintaining a constant value of argon flow. The thickness of TiN coatings was about 0.4 μm. For assessing the effect of magnetron discharge plasma on the formation of TiN films and their physical and mechanical properties, the distance between the target and the substrate (L<sub>s-c</sub>) was chosen 100 and 50 mm.

The performance of MRS depending on the flow rate of nitrogen Q(N<sub>2</sub>) at different values of L<sub>s-c</sub> is shown in Figure 6.

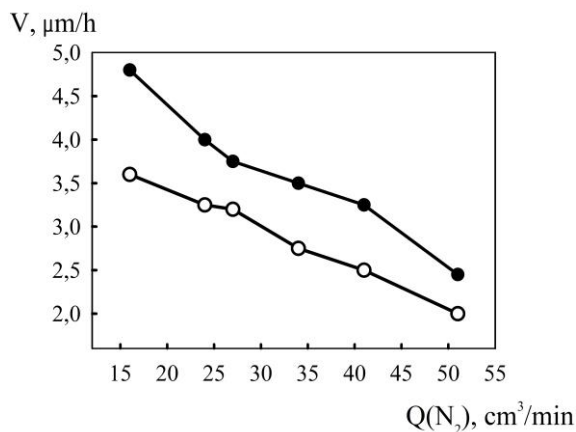


Figure 6 – The influence of nitrogen flow rate Q(N<sub>2</sub>) on the productivity of the process of applying Ti coatings TiN at different distances between the plane of the target and the substrate L<sub>s-c</sub>: ● – 100 mm; ○ – 50 mm

Due to the imbalance of the magnetic field of the magnetron, the growing coating is digested by the ionic flow of the plasma discharge. There is a decrease in the deposition rate of TiN by an average of 20 % when approaching the plane of the substrate to the target by 50 mm.

The decrease in system performance with increasing Q(N<sub>2</sub>) is due to the “poisoning” of the titanium cathode with a layer of TiN and a lower coefficient of sputtering of the target with nitrogen ions.

Photomicrographs of the surface and cross-section of thin TiN coatings applied by reactive magnetron sputtering are shown in Figure 7.

The photomicrograph of the surface (Figures 7 a, b) shows a relatively high homogeneity of the studied coatings (no punctures).

As an illustration, Figure 7 c shows a fractogram of TiN coating. You can see that the obtained coating has a columnar structure inherent in the ion-plasma coating. A study of the surface morphology of coatings showed that they have good quality.

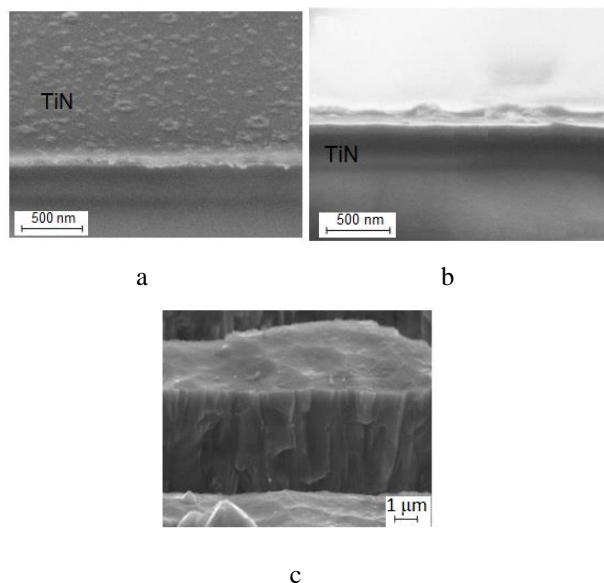
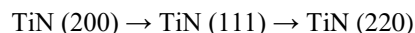


Figure 7 – Microstructure of thin TiN coatings: surface (a), cross-section (b), and fractogram of fracture for TiN coating obtained by magnetron sputtering (c)

According to the results of X-ray diffraction studies of experimental samples, we have a TiN coating with a polycrystalline structure and orientation in the crystallographic directions (111), (200), (220). Peaks of intensities (311) and (222) are weakly shown.

The orientation of the growth of coatings in one of the crystallographic directions is associated with changes in the energy of scattered particles and ions falling on the substrate. Increasing the energy of the generated particles stimulates the formation of titanium nitride coatings according to the following scheme:



The crystallographic direction (220) becomes predominant in the nitride coating when the specific energy losses of the precipitated particles become significant. TiN coatings obtained at L<sub>s-c</sub> = 100 mm are more oriented along the axes (111) and (200). When the substrate approaches the target (50 mm), intensity peaks become significant (220).

The formation of TiN coatings with a predominant orientation (111) occurs in a gaseous medium N<sub>2</sub>, without the supply of Ar. Changing the flow rate of nitrogen into the working chamber stimulates a decrease in the intensity of reflexes (200) and (220) for the distance

“target-substrate”  $L_{s-c}$  100 and 50 mm, respectively. From the X-ray diffraction data, the values of the parameters of the crystal lattice of the experimental samples 4.22–4.25 Å correlate well with the data for work [14].

There is no evident dependence of the mechanical properties of the coatings on the  $N_2$  flux. Reducing the “target – substrate” distance reduces the mechanical properties of the coatings.

#### 4.1.2 Study of coatings based on AlN

AlN coatings were formed by magnetron jet spraying cylindrical targets with Al in a gas mixture of  $Ar^+$  40–60 % vol.  $N_2$  at a pressure of 0.6–0.9 Pa. The working gases were fed separately ( $Ar$  – in the spray area of the target,  $N_2$  – in the substrate area) and as a mixture.

The obtained coatings have a fibrous (columnar) structure, while AlN fibers have a hexagonal lattice. Crystalline phases of AlN coating are detected using electron microscopy.

The disadvantage of the magnetron sputtering method is the presence of drip phase in coatings, which leads to deterioration of the integrity and corrosion properties (Figure 8) of the scanning electron microscope (SEM).

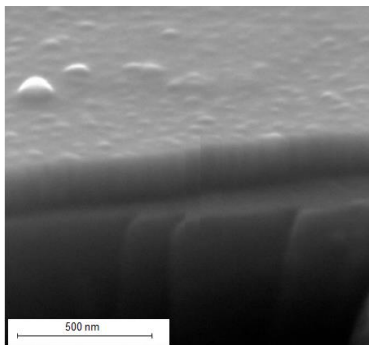


Figure 8 – Structure (SEM) of the surface of the AlN coating obtained by magnetron sputtering on a polished substrate of stainless steel P6M5

By varying the pressure  $N_2$  in the vacuum chamber, it is possible to change the color of the treated surface to some extent. Arc evaporation forms coatings with a smooth surface corresponding to the roughness of the substrate.

At low rates of deposition of coatings, as well as the absence of electrical displacement on the substrate holders, formed coatings consisting of equiaxed disordered crystallites (Figure 9 a). Changing the synthesis conditions promotes the formation of crystallites in the form of fibers.

The structure of the growth and chip surface of the AlN film obtained on a substrate of P6M5 steel shows (with appropriate changes in the parameters of the deposition process) the transition of the lamellar structure to fibrous and fine-grained with equilibrium grains (Figure 9 b).

After studying the coatings of TiN and AlN, it was concluded that they have certain disadvantages that can be significantly reduced by obtaining compounds based on components of Ti-Al-N.

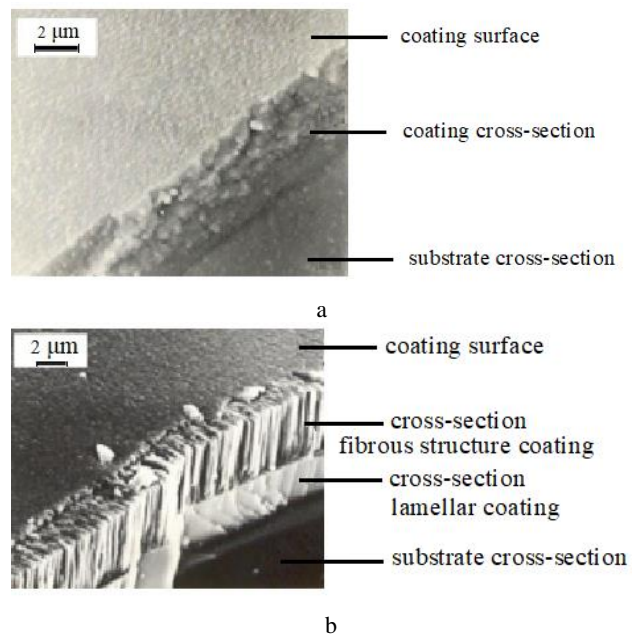


Figure 9 – Structure (SEM) of AlN coatings formed by magnetron sputtering

#### 4.1.3 Structure and properties of coatings based on TiAlN

In the manufacture of tools from reinforcing coatings, the main focus is on the TiAlN compound, due to good performance properties, which are characterized by obtaining a wide range of stoichiometric composition and reproducibility of their production, so the application of carbonitride coatings (e.g., TiCN) is much more difficult to control. Nitride coatings are the most technological in production, and it can be argued that TiAlN and its modifications (e.g., AlTiN, TiAlCrN, TiAlCrYN) have become the flagship of the global coatings industry and are widely used as completely self-sufficient coatings and in various multilayer compositions. (TiAl)N have unique performance properties: high hardness at high temperatures in the cutting contact zone; thermal and chemical resistance, high elasticity; low thermal conductivity. One of the essential advantages of (TiAl)N coating is its tendency to oxidize and form relatively stable surface oxide films. You can increase the wear resistance (TiAl) of N coatings during cutting by grinding the grain to the nanoscale level (grain size less than 100 nm). This is possible by doping (TiAl)N coatings on the tool material by magnetic arc filtration. The coating is used for processing titanium and nickel alloys, stainless steel, hardened materials, cast steel, and plastics. The temperature stability of the coating determines the possibility of its use in high-speed and emulsion-free processing of materials.

The results of studies of the crystal structure of samples by transmission electron microscopy (TEM) showed the following: increasing the value of the negative voltage applied to the substrate during deposition of wear-resistant coatings from the target  $Ti_{0.5}Al_{0.5}$  in the N atmosphere increases the concentration of defects and dislocations in the structure. The



dependence of the average grain size  $D$  on the value of the negative voltage  $V_S$  applied to the substrate in the condensation process of the samples is shown in Table 2.

Table 2 – Dependence of the average grain size  $d$  on the value of the negative voltage applied to the substrate

$V_S$ , V	0	50	75	100	125	150	250
$d$ , nm	105	95	60	55	45	42	40

As can be seen from Table 1, the value of  $d$  decreases from 105 nm to 55 nm when changing the value of the negative voltage  $V_S$  from 0 to 100 V. Further increase in the negative voltage on the substrate during condensation does not significantly affect the grain size change and even at  $V_S = 250$  V. The grain size remains about 40 nm.

Figures 10 a, b show the surface microstructures and the cross-section for single-layer TiAlN coatings, respectively.

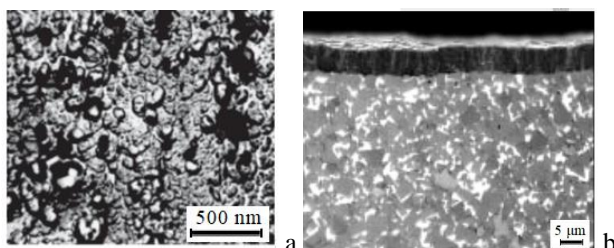


Figure 10 – Surface (a) and cross-section (b) of a plate of high-speed steel P6M5 with TiAlN coatings (5  $\mu$ m)

Energy-dispersion spectra, typical for all series of TiAlN coatings on silicon plate substrates and characterizing their stoichiometry, are shown in Figure 11. The concentration of Al varies from 34 to 35 %, the concentration of Ti varies from 52 to 54 %, and the concentration of N is within 10 %.

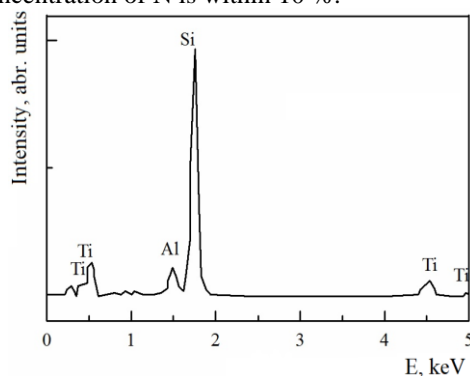


Figure 11 – Energy dispersion spectra and the elemental composition of TiAlN coating determined by them

The main components of the coatings obtained in a mixture of argon and nitrogen are TiN, AlN, and nitride phase of variable composition  $(Al_xTi_{1-x})N$  with cubic lattices based on TiN. The concentration of the coating components depends on both the concentration of the metal components Ti and Al and the pressure of the reaction gas composition present in the deposition zone. The average size of the formed crystallites: for TiN – 10 nm,  $(Al_xTi_{1-x})N$  – 5 nm, and AlN – 10–100 nm. Similar results were obtained by the authors [23].

Figure 12 shows the dependencies of hardness on the

applied negative stress on the substrates.

As shown in Figure 8, the higher the electric field on the substrates, the greater the hardness of the coating  $[Ti(Al)]_xN_{1-x}$ .

This is due to the phase transition from hcp-AlN (MSN  $AlN_x$ ) to c-TiN (cubic-TiN) and can be explained by the high level of compressive stresses (5–6 GPa), which is confirmed by other authors [11].

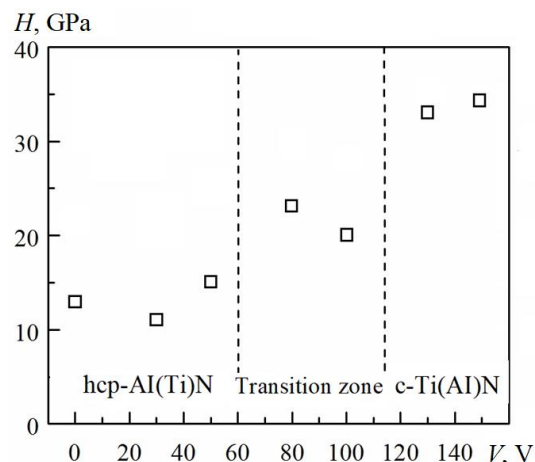


Figure 12 – Dependence of hardness on the applied shear stress on P6M5 steel substrates during condensation

The lowest values of hardness are observed when  $AlN_x$  structure crystallizes in coatings. By increasing the value of the negative stress on the substrate, in this case, you can achieve a slight increase in hardness due to the slow growth of compressive stresses in the sample and reducing the grain size, respectively, as in work [11].

We can conclude that by regulating the energy and flux density of metal ions of alloying elements, it is possible to control the processes of microstructure formation and the hardness of nanocrystalline coatings.

#### 4.1.4 Structure and properties of TiAlSiN coatings

TiAlN coating is superior to TiN coating in hardness, wear resistance, and especially oxidation resistance. The cutting temperature in the modern high-speed cutting industry can reach 1 000  $^{\circ}C$ , which can cause oxidation and reduced hardness of TiAlN coatings. Due to this, nanocomposite coatings based on Ti, Al, Si, and N are now increasingly used for cutting tools. Compared to traditional durable TiN, AlN, or TiAlN coatings, the TiAlSiN coating, which forms the structure of the amorphous  $Si_3N_4$  shell for the TiAlN nanocrystalline coating, has attracted worldwide attention due to its enhanced properties as a complex coating for multilayer coatings and multicars [3, 6, 20, 24-29].

Ti-Al-Si-N coatings were prepared by magnetron sputtering using different bias voltages applied to 30 V and 130 V substrates and deposition energy. In this way, the microstructures and mechanical properties of the coatings can be adjusted, such as grain size, orientation, and hardness.

As the bias voltage increased, the composition of the coatings remained almost unchanged, maintaining the composition of  $Ti_{0.19}Al_{0.25}Si_{0.05}N_{0.51}$ .

Figure 13 presents X-ray diffraction patterns of sprayed coatings Ti-Al-Si-N at bias voltages of  $-30$  V and  $-130$  V. We see cubic TiAlN (111), (200), (220), and diffraction peaks of wurtzite AlN (002) in coatings, except for the peak of the Si substrate. However, the diffraction peaks of  $SiN_x$  phases in coatings are not observed. This can be explained because silicon can exist as an amorphous silicon nitride, which corresponds to the work [28-29].

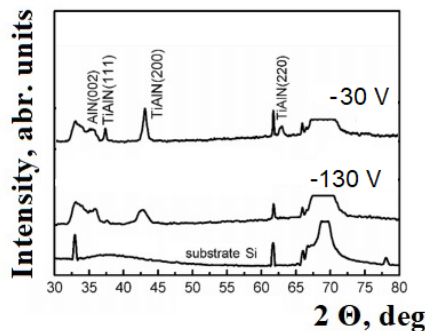


Figure 13 – X-ray diffraction patterns of Ti-Al-Si-N coatings deposited at different displacements stresses applied to the substrate

As shown in the cross-sectional images in Figure 14, obtained on a silicon substrate, the thickness of the coating Ti-Al-Si-N is about  $1.8$  to  $1.9$   $\mu m$ . As can be seen from the figure, there were no voids in the coatings, and the microstructure was dense. At a displacement of  $-30$  V, very thin columns of the morphology of zone I can be seen in the model, which ended with the structure of the surface zone. At a displacement of  $-130$  V, the sections were flat and did not have such a pronounced columnar structure.

According to diffraction measurements, the nanolayer TiAlSiN coating consists of crystalline phases of solid solution-TiN and amorphous phase  $Si_3N_4$ . TEM analysis showed that TiSiN layers block the growth of TiAlN crystallites, which are balanced and about  $5$  nm in size. As a result, the TiAlSiN coating shows high hardness ( $H = 37$  GPa), which is explained by the limited dislocation activity in small crystals and the suppression of sliding grain boundaries.

The diffraction peaks of TiAlN coating are shifted to higher diffraction angles compared to stoichiometric TiN, which indicates a decrease in the lattice parameter ( $0.418$  nm compared to  $0.424$  for pure TiN) [24-25].

No signals were received from the crystalline silicon nitride or titanium silicide phase, indicating that the Si-containing phase is amorphous or too thin.

In Figure 15, SEM images of the cross-section of TiAlSiN coating on P6M5 steel are shown. The coating adheres well to the substrate. It is uniform in thickness and has no structural defects such as cracks and voids. Columnar growth is observed in the coating.

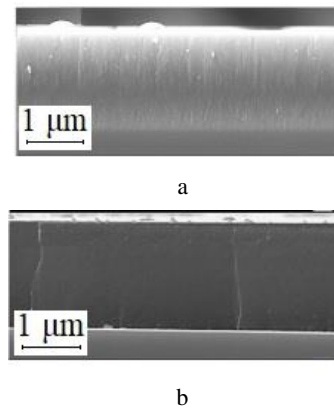


Figure 14 – Image in cross-section of Ti-Al-Si-N coatings applied at different bias voltages  $-30$  V (a) and  $-130$  V (b)

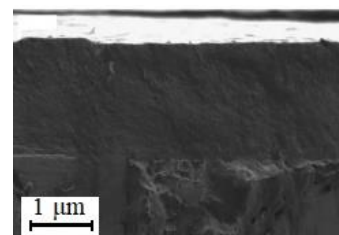


Figure 15 – Photomicrograph of cross-section, showing the structure of TiAlSiN coating on P6M5 steel

The  $H$  hardness of Ti-Al-Si-N nanocomposites varied at shear stresses  $-30$  V and  $-130$  V. With increasing shear stress, the hardness increased from  $32$  GPa to  $37$  GPa, and at a voltage of  $-130$  V, the coating showed the most significant hardness. High values of TiAlSiN coating hardness since it has high resistance to cracking.

The increase in mechanical properties is mainly due to increasing the residual stress during compression and reducing the grain size in the Ti-Al-Si-N coating. TiAl-Si-N coatings with high hardness values and dense macrostructure showed increased resistance to cracking and wear, which is in agreement with the literature [30].

It is well known that hardness increases with decreasing grain size, which can be explained by an increase in areas of grain boundaries that interfere with dislocation movements in the form of dislocation accumulations (Hall-Patch effect) [24-25].

Ti-Al-Si-N nanocomposite coating maintained high hardness at annealing temperature up to  $1000$  °C and has increased resistance to oxidation up to  $1115$  °C.

#### 4.2 Determination of mechanical properties of coatings

The hardness of the material characterizes the degree of its ability to resist plastic deformation, and the fracture toughness is a measure of its resistance to crack propagation and fracture. With the optimal combination of hardness and resistance to destruction, the service life of the coating is determined by its wear.

Mechanical properties and characteristics of wear-resistant coatings are given in Table 3.

P6M5 steel samples and samples of the same steel but coated were tested for wear for  $60$  and  $120$  minutes. The test results are shown in Table 4.



Table 3 – Mechanical properties and characteristics of wear-resistant coatings

Coating	Color	Hardness $H\mu$ , GPa	Thickness, $\mu\text{m}$	Maximum temperature, $^{\circ}\text{C}$
TiN	gold	20-24	2–7	600
AlN	gray	12-16	2-5	600
AlTiN	gray-purple	32	1-3	800
TiAlN	purple-black	35	1-7	900
TiAlSiN	brown	32-37	1-4	1 100
Uncoated steel P6M5	gray	6-9	N/A	N/A

Table 4 - Wear intensity of TiN and TiAlN coatings on P6M5 steel samples

Coating	Wear intensity I, $10^{-7}\text{kg/m}^2$	
	after 60 min	after 120 min
TiN	7.6	18.0
AlN	8.8	19.9
TiAlN	3.8	9.0
TiAlSiN	4.8	9.3
Uncoa-ted steel P6M5	25.0	54.0

As a result, it was found that the wear intensity of steel after 120 minutes of uncoated tests is 5–6 times higher than with TiAlN and TiAlSiN coating, 3 times - than with TiN and AlN coatings.

When comparing the productivity during drilling with a P6M5 steel tool, it is shown that the number of holes drilled with an uncoated drill is 47 when using a drill with TiN coating – 88 holes, when using a drill with TiAlN coating – 246 holes. That is, coating increases the productivity of the tool by 2–5 times, which is confirmed by the work of other authors [3, 6, 13-16, 20].

The obtained coatings TiN, AlN, TiAlN, AlTiN, and TiAlSiN are layers that are very difficult to erase, well protect the tool from surface wear, reduce energy consumption, increase tool reliability and redness.

Cutting properties of the protected tool, 1.5–5.0 times higher than the tool without covering.

## 5 Conclusions

The structural-phase and mechanical properties of wear-resistant coatings based on Ti, Al, Si, and N obtained by magnetron sputtering were analyzed.

By varying the synthesis conditions, you can change the coatings' phase composition and structure. Functional properties largely depend on the composition and morphology of coatings.

The advantage of TiN is good elasticity and adhesion. According to X-ray diffraction data, the TiN samples obtained by us have a polycrystalline structure (111), (200), and (220).

The surface morphology strongly depends on the distance “target – substrate” TiN coatings have a crystalline structure of NaCl and an average lattice parameter of  $4.24\text{ \AA}$ , and the structure consists of covalent, metallic, and ionic bonds. The covalent bond explains the high hardness of up to about 20 GPa.

Resistance to loads of textured AlN films is provided both due to the high concentration in the protective coating of crystalline AlN. When the grains of the crystalline phase of the protective coating is oriented parallel to the direction of mechanical loads' action, the coating's integrity is preserved under the influence of shock loads.

TiAlN material is used in various areas, such as protective and wear-resistant coatings, diffusion barriers, and optical devices. The hardness of AlTiN and TiAlN reaches 32–35 GPa depending on the deposition conditions and composition. TiAlN has excellent resistance to high temperatures. Suitable for high-speed processing, it can be used without coolant.

Ti-Al-Si-N nanocomposite coatings were obtained by magnetron sputtering at bias voltages of –30 and –130 V. With increasing shear stress of the substrate, Ti-Al-Si-N coatings had a decrease in (200) -preferential orientation: dense columnar the structure (zone I) of Ti-Al-Si-N coatings gradually evolved into an uncharacteristic and flat structure. With increasing shear stress, the hardness increased from 32 GPa to 37 GPa, i.e., at a displacement of –130 V, the coatings had the highest hardness. Ti-Al-Si-N nanocomposite coating maintained high hardness at annealing temperature up to 1 000  $^{\circ}\text{C}$  and has increased resistance to oxidation up to 1 115  $^{\circ}\text{C}$ . TiAlSiN coatings are characterized by high hardness values, which means high viscosity of their destruction, high resistance to cracking, and should be helpful in tribological applications.

In the obtained magnetron sputtering coatings on samples of P6M5 steel, the microhardness of coatings is TiN – 20–24 GPa, AlN – up to 16 GPa, TiAlN – up to 35 GPa, AlTiN – up to 32 GPa, TiAlSiN – 32–37, while the microhardness of the substrate itself steel P6M5 – 6–9 GPa. The microhardness of two-element TiAlN coatings applied to the tool base is 1.5 to 1.9 times higher than the microhardness of one-element TiN and AlN coatings.

It was found that the intensity of steel wear after 120 minutes of uncoated tests is 6 times higher than with AlTiN and TiAlN coating, 3 times higher than with TiN coating.

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