Influence of the Thickness of Multilayer Composite Nano-Structured Coating Ti–TiN–(Ti,Al,Si)N on the Tool Life of Metal-Cutting Tools and the Nature of Wear

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Abstract: This article discusses the influence of the thickness of a nano-structured wear-resistant layer of the Ti–TiN–(Ti,Al,Si)N multilayer composite coating on its mechanical and performance properties. The study was focused on the coatings with the following thicknesses of its wear-resistant layers: 2, 3.5, 5, 7, 11, and 15 µm. The relation between the thickness of a wear-resistant layer and the time of its deposition was investigated, and the effect of the above thickness on hardness and wear resistance in scratch testing was considered. Cutting tests were conducted in turning steel C45 with carbide inserts with the coatings under study at various cutting speeds (v_с = 250, 300 and 350 m/min). The study found the value of thickness of wear-resistant layer providing the longest tool life at various cutting speeds. The differences in the nature of wear for the coatings with various thicknesses of wear-resistant layers were considered.

Keywords: wear-resistant coatings; coating thickness; wear; tool life

1. Introduction

The challenge to choose the optimal thickness for coatings of different compositions is considered in several studies devoted to the investigation of the properties of physical vapor deposition (PVD) coatings. In Reference [1], Klocke et al. found that the coating with the maximum thickness provided the longest metal-cutting tool life. Meanwhile, it is indicated that, at the deposition of a single layer of coating, the grains increase with an increase in the thickness of the coating [2]. This fact is related to the mobility of adatoms (adsorbed atoms) during the deposition of a coating, since the deposited coating becomes gradually insulating. In other words, it acts as a barrier to a plasma flow and reduces the energy of bombardment of a coating surface with particles. As a coating grows, the adatoms move around the surface until they find a potential “crater” for embedding, usually a defect or a position near the steps of a growing crystal on the surface (Figure 1) [3].
Accordingly, with the increase in the thickness of a single layer of coating, its superficial hardness is reduced [1]. It can also be assumed that thin coatings have a higher mechanical strength than thicker coatings. It is shown that nominal superficial hardness, superficial yield, and maximum superficial hardness decrease with an increase in coating thickness [1].

There are several studies [4–25] that consider the challenge of determining the optimal thickness for the coatings of various compositions for different cutting conditions. However, almost all of the above-mentioned studies solve the challenge in relation to monolithic single-layer coatings. In particular, Vereshchaka [4] considered the effect of the thickness of the TiN, TiC, (Ti,Cr)N coatings with thicknesses of 1–14 μm on the oxidation characteristic of carbide tools. It was found that, when the coating thickness was less than 1 μm, its effect on the oxidation process was minimal, and when the coating thickness was 3 μm or more, the oxidation resistance of the tool increased significantly. Meanwhile, when heated up to 900–1200 °C, coatings failed on the samples with coating thicknesses over 10 μm. It was also found [4] that thicker coatings proved to be more efficient in turning as the cutting speed increased. This can be explained by the fact that high temperatures sharply initiate corrosion–oxidative and diffusion processes, and, consequently, a barrier role of the coating increases. Experimental studies found [4] that, for a number of PVD coatings, the optimum thickness in turning steel and cast iron stayed within a range of 6–8 μm and in milling at 4–6 μm.

Meanwhile, Posti et al. [5] noted that a tool with a coating thickness of 2–3 μm showed the longest tool life in turning steels. Methods of mathematical modeling [6] were also used, and they made it possible to determine the optimum thickness of the coating for turning steel at 5.42–5.74 μm. Bouzakis et al. [7,8] found that, in milling steel with carbide tools with the (Ti46Al54)N coating, the longest tool life was shown by a tool with the greatest thickness (10 μm) in coating. Meanwhile, Tuffy et al. [9] studied the effect of the thicknesses of the TiN, TiAlN coatings on the tool life of carbide tools in turning of steel, and found that the longest tool life was registered for a tool with the coating thickness of 3.5 μm.

The worst properties of the coatings with greater thickness can be explained, in particular, by the data obtained by Ma et al. [10], showing that, with an increase in the coating thickness, internal intercrystalline shear stresses also increased. It was also found [10] that inclined cracking was typical for thicker coatings. It is assumed that an increase in the strength of the grain boundaries occurred simultaneously with the absence of a direct crack path along the grain boundaries through the thicker coatings due to a more equiaxed grain structure.

The dependence of the deposition time on the coating thickness was also investigated. Avelar-Batista et al. [11] considered the CrN coating with different coating thicknesses and found that, in all cases, there was a linear relationship between the deposition time and coating thickness. Moreover, with an increase in the coating thickness, the deposition rate of coating did not change.

There are studies analyzing the optimal thickness of nano-structured coatings. In particular, Skordaris et al. [12] found that a tool with the nano-structured Ti46Al54N–TiN coating showed a significantly longer tool life (due to good crack resistance). A sample with the thicker coating (8 μm) showed a longer tool life in nano-impact tests and the longest tool life in milling. The effect of the thicknesses of nano-structured coatings of different compositions on their wear resistance
was also considered [13–20] (Ti–TiN–(TiCrAl)N and ZrN–(Zr,Al,Si)N [13,14], Zr–(Zr,Cr)N–CrN and Ti–TiN–(Ti,Cr,Al)N [15], Ti–TiCN–(Ti,Al)CN and Ti–TiCN–(Ti,Al,Cr)CN [16], Ti–TiN–(TiCrAl)N and Ti–TiN–(Ti,Al,Si)N [17], and (Ti,Al)N–(Zr,Nb)N–CrN [18–20]). It was also found that layered nano-structured coatings could have significant advantages compared to single-layer and monolithic coatings. The effect of the coating thickness on its wear pattern and cracking and on the features of cracking and failure of nano-structured coatings for cutting tools were considered by Bouzakis et al. [21,22], Skordaris et al. [23], and others [24,25].

Nano-structured coatings are characterized by a number of properties that distinguish them from monolithic coatings of the traditional type. Kim et al. [26] considered the nanolayer CrN–(Cr,Al)N coating with the thicknesses of binary monolayers of \( \lambda = 4–44 \) nm. It was found that, with a decrease in the thickness of nanolayers, a more ordered orientation of the elementary layers in the crystalline domains was observed. The best parameters (hardness, resistance to plastic deformation, low friction coefficient, and wear resistance) were registered for the coating with \( \lambda = 7.4 \) nm. Fox-Rabinovich et al. [27] considered the properties of the nano-structured (Al,Ti)N and (Ti,Al,Cr)N coatings and determined the areas of their preferred use.

Nano-structured coatings have several advantages. Biksa et al. [28] studied the properties of a series of the (Al,Ti)N-based nano-structured coatings and found that a tool with such coatings demonstrated noticeably higher tool life compared to a tool with the single-layer (Al,Ti)N coatings. Liang et al. [29] found a significant improvement in properties of the TiN–(Ti,Al)N coating, such as the strength of the adhesive bond with the substrate, the microhardness in combination with the elastic modulus, and the resistance to oxidation in the presence of nano-structure, when the optimal value of \( \lambda_{BD} \) was 20 nm. A number of studies have shown that the introduction of an insignificant amount of Si (3–5 at.\%) into the composition of such coatings makes it possible to further improve their performance properties by inhibiting grain growth and forming a nanocrystalline structure [30–34].

In [35–38] by Veprek research group, much attention was paid to the study of nano-structured coatings. In particular, it was found that nano-structured coatings provided an increase in hardness, but the effect was limited by grain boundary shear, when crystallite sizes decreased to 10–15 nm, (the size is usually referred to as “strongest size”). A further decrease in grain sizes leads to a decrease in hardness of the coating. However, in the case of low-energy grain boundaries (for example, in the case of stacking faults and twins or in the case of one-monolayer thin sharp grain boundaries, formed when Si\(_3\)N\(_4\) is introduced to the coating composition), “strongest size” can be reduced to several nanometers. Meanwhile, the hardness of such nanocomposite coating can reach 100 GPa [37,38].

After the analysis of the available results of the studies [1–38], the following conclusions may be drawn:

- When the thickness of the coating increases, its barrier properties increase with respect to diffusion and oxidation processes, which is especially important for high cutting speeds.
- Meanwhile, when the thickness of a single-layer coating increases, its grains and inner intercrystalline stresses increase. Due to the long-term thermal effect on the tool substrate in the deposition of thick coatings, its strength and surface hardness may decrease.
- The use of single-layer coatings with thicknesses of less than 2 \( \mu \)m and over 10 \( \mu \)m is not reasonable because of the low performance properties.
- For the coatings of different compositions and architecture, there are different values of the optimal thicknesses that may also differ significantly under different cutting conditions.
- Coatings based on the TiN–(Ti,Al,Si)N system with nanolayer architecture may be effectively used to improve the tool life of a metal-cutting tool in cutting various metals and alloys [30–34].

Thus, this study was aimed to consider the specifics of deposition of multilayer nano-structured coatings based on the three-layer Ti–TiN–(Ti,Al,Si)N system with different thicknesses of wear-resistant layers. Another aim of the study was to investigate the processes of wear and the failure of tools with the coatings under study in cutting various materials (structural steel C45 and at various cutting speeds).
2. Materials and Methods

To deposit the coatings, a VIT-2 unit was used (developed by IKTI RAN – MSTU STANKIN). One of the specific features of the VIT-2 unit is the use of arc evaporator with filtration of vapor-ion flow (FCVAD) [13–20], which makes it possible to obtain coatings with clearly defined boundaries of nanolayers. Coatings with three-layer architecture proved in [18–20] were deposited, and the Hume-Rothery rules [39] were followed to select the proportion of elements in the coating composition. Considering the results of the earlier studies [40–46], the composition of the wear-resistant (Ti_{0.70},Al_{0.25},Si_{0.05})N coating was chosen. This composition provides sufficient hardness and wear resistance, in combination with good adhesion to the substrate and resistance to brittle fracture.

The parameters used at each stage of the deposition process of coatings are shown in Table 1.

| Process                              | $p_N$ (Pa) | $U$ (V) | $I_{Al, Si}$ (A) | $I_{Ti}$ (A) |
|--------------------------------------|------------|---------|-----------------|-------------|
| Pumping and heating of vacuum chamber| 0.06       | 20      | 120             | 65          |
| Heating and cleaning products with gaseous plasma | 2.0        | 100DC/900 ACf = 10 kHz, 2:1 | 80 | – |
| Deposition of coating               | 0.36       | –800 DC | 160             | 55          |
| Cooling of products                 | 0.06       | –       | –               | –           |

Note: $I_{Ti}$ = current of titanium cathode, $I_{Al, Si}$ = current of Al–Si cathode, $p_N$ = gas pressure in chamber, $U$ = voltage on substrate.

The time of deposition of wear-resistant coating layers are shown in Table 2.

| Sample Designation | S2 | S3 | S4 | S5 | S6 | S7 |
|--------------------|----|----|----|----|----|----|
| Deposition time, min | 15 | 30 | 45 | 60 | 90 | 120 |

An uncoated carbide tool and a carbide tool with a reference coating of TiN, which was deposited using the commercial technology of arc PVD, were used as objects for the comparative studies of tool life.

Coatings of equal thickness were deposited on all samples in a single technological cycle to ensure the correctness of the research. Inserts made of commercial cemented carbide (P group by ISO 513:2012 [47]; 79% WC, 15% TiC, 6% Co; KZTS, Russia) were used as samples.

For microstructural studies of samples of carbide substrates with coatings, a scanning electron microscope (SEM) FEI Quanta 600 FEG (Materials & Structural Analysis Division, Hillsboro, OR, USA) with energy-dispersive X-ray spectroscopy (EDS) equipment was used. The studies of chemical composition were conducted using the same SEM. To perform the X-ray spectroscopy microanalysis, the study used characteristic X-ray emissions resulting from the electron bombardment of a sample.

The X-ray diffraction analysis (XRD) was performed with a PANalytical Empyrean diffractometer (Malvern Panalytical Ltd, Malvern, UK) with Cu Kα radiation. The XRD patterns were collected using Bragg-Brentano and parallel beam geometry. The XRD patterns were analyzed using PANalytical High Score Plus software and the ICDD PDF-2 and COD databases [48]. The studies of nano-structure involved a high-resolution transmission electron microscope (TEM) JEM 2100, manufactured by JEOL Company, Tokyo, Japan. The hardness (HV) of coatings was determined by measuring the indentation at low loads according to the method by Oliver and Pharr [49], which was carried out on a microindentometer microhardness tester (CSM Instruments, Needham, MA, USA) at a fixed load of 100 mN.

The tests of coating adhesion strength were carried out on a Nanovea M1 scratch-test tester Micro Scratch, Nanovea, Irvine, CA, USA) according to Reference [50]. A Rockwell C Diamond indenter was used in the form of a diamond cone with a vertex radius of $R = 100$ µm and cone angle of 120°.
SNUN ISO 1832:2012 [51] inserts identical to those used in the studies of cutting properties were used as a substrate in scratch testing.

A CU 500 MRD (ZMM-BULGARIA HOLDING, Sofia, Bulgaria) lathe was used while testing the cutting properties of the tools with the coatings under study. The tests were carried out in longitudinal turning of steel C45 (HB 200). Cutters with cemented carbide SNUN ISO 1832:2012 [51] inserts were used as cutting tools. The following cutting geometry was used: $\gamma = -7^\circ$, $\alpha = 7^\circ$, $K = 45^\circ$, $\lambda = 0^\circ$, and $R = 0.8$ mm.

Based on the available recommendations and results obtained earlier [52], the following cutting modes were used: $f = 0.2$ mm/rev; $a_p = 1.0$ mm; and $v_c = 250, 300, \text{and} 350$ m/min;

Flank wear-land values ($V_Bc$) were measured with an optical microscope (MBS-10, LZOS, Lytkarino, Russia) as the arithmetic mean of four to five tests, and $V_Bc = 0.4$ mm was taken as the failure criteria.

3. Results and Discussion

3.1. Microstructure, Thickness, Elemental, and Phase Composition of Coatings

The investigations of the microstructure of the coatings under study and their designations are shown in Figures 2 and 3. Given that the coating thickness has a spread of ±0.4 μm, Figure 3 shows the average thickness, rounded to the nearest integer value. The ratio of deposition time and thickness of the wear-resistant layer represents a virtually ideal linear relationship (Figure 4).

The outer wear-resistant layer of the coatings under study is multilayered and nano-structured (except for TiN coating, see Figure 2) with an average thickness of 43–85 nm for the nanolayers. Meanwhile, the number of nanolayers is within the range of 39–180.

Let us consider the coating deposition pattern using three cathode systems: Two cathode systems of the traditional type with installed Ti cathodes (99.8 at.%)(2 and 3 in Figure 5a), and a system with droplet phase filtration [13–15] with the installed Al–Si cathode (94.3 at.% + Si 5.0 at.%) (1 in Figure 5a). In the case of a planetary rotation (4) of a sample (5) in such a system (6), there is a complex pattern of coating formation. It is important to note that, in almost all areas along the sample motion trajectory, the flows of Ti and Al + Si interfuse with a smooth gradient change in their ratio. Accordingly, over the entire volume of the coating, it is possible to predict the presence in the composition of both nitride Ti and nitride Al, as well as in a single-phase solid solution (Ti,Al,Si)N. Only the ratio of these elements will change.

![Figure 2. Coating. Average thickness of the coating is 4.0 μm (S1).](image-url)
Figure 3. Thickness and elemental composition of coatings (S2–S7) under study.
Let us consider the coating deposition pattern using three cathode systems: Two cathode systems of the traditional type with installed Ti cathodes (99.8 at.%) (2 and 3 in Figure 5a), and a system with droplet phase filtration [13–15] with the installed Al–Si cathode (94.3 at.% + Si 5.0 at.%)(1 in Figure 5a). In the case of a planetary rotation (4) of a sample (5) in such a system (6), there is a complex pattern of coating formation. It is important to note that, in almost all areas along the sample motion trajectory, the flows of Ti and Al + Si interfuse with a smooth gradient change in their ratio. Accordingly, over the entire volume of the coating, it is possible to predict the presence in the composition of both nitride Ti and nitride Al, as well as in a single-phase solid solution (Ti,Al,Si)N. Only the ratio of these elements will change.

Thus, the nanolayer structure of the coating has a rather complicated structure. The structure contains clearly expressed nanolayers noticeable in Figure 3. Each such binary nanolayer is formed when a sample gradually passes through the zone of the predominant Al content (narrower dark area in SEM images) and the zone of the predominant Ti content (thicker light area in SEM images). However, in passages through the Al zone, the planetary gear manages to make several turns, and several nanolayers of various thickness are formed (hereinafter, sub-nanolayers; see Figure 5, in TEM images, the sub-nanolayers with the high Al content are light, in contrast). Figure 6 presents the TEM image of the cross cut of sample S4. It is possible to note nanolayers with high Al content [53] and a total thickness of 20–30 nm. In turn, each of the nanolayers consists of 3–6 sub-nanolayers with a thickness of 2–6 nm. The number of sub-nanolayers in a layer depends on the number of turns of the planetary gear in the passage of the Al-rich area.
Let us consider in more detail the structure of nanolayers of the coating under study. Figure 7a shows the multidirectional orientation of the crystalline planes in various crystallites of the coating. While considering the boundaries of the zones with an equivalent orientation of crystalline planes, it is possible to determine the approximate boundaries of the crystallites. In Figure 7, interplanar distance can correspond to \( d_{111} = 2.448 \text{ Å} \) for the TiN (Fm3m) phase and to \( d_{002} = 2.485 \text{ Å} \) for the AlN (P6.3mc) phase.

![Figure 6. Nano-structure of the wear-resistant (Ti,Al,Si)N layer of the coating (at S4) (TEM).](image)

![Figure 7. Nano-structure of the wear-resistant (Ti,Al,Si)N layer of the coating (sample S4) (TEM). General view of the nanolayer with a high aluminum content (light contrast on TEM) (a); area A2 (b); area B2 (c); crystalline planes (d).](image)
During the study of an area of a coating sub-nanolayer with high Al content (Figure 8), it is also possible to notice a difference in the orientation of the atomic layers, and thus determine the boundary of the crystallites. In this case, the measured distance between the atomic layers is about 0.265 nm, which corresponds to AlN \( (d_{100} = 2.685 \text{ Å}, \text{P6.3mc}) \).

![Figure 8. The wear-resistant (Ti,Al,Si)N layer of the coating (at S4) (TEM).](image)

As known, TiN has a cubic side-centered NaCl-type lattice, the space group Fm3m, with the period of \( a = 0.423 \text{ nm} \) (for stoichiometric films, the value \( a = 0.425 \text{ nm} \) is most often given and for stoichiometric massive TiN, \( a = 0.424 \text{ nm} \)). The AlN has the period of \( a = 0.311 \text{ nm} \) for the structure of h-wurtzite and \( a = 0.438 \text{ nm} \) for the structure of c-sphalerite [54]. It was noted [55] that the parameter \( a \) could increase with an increase in nitrogen content and with an increase in internal stresses [56]. Following the analysis of the obtained results, it is difficult to unambiguously determine the phases with close interplanar distances, especially in the presence of stresses in the solid solution of AlN, TiN, (Ti,Al,Si)N.

Under deposition conditions, unbalanced (metastable) phases are formed in the coatings. As a rule, they are single-phase solid solutions of (Ti,Al,Si)N with cubic or wurtzite crystalline structure, depending on the Al content. Then, there is a phase transition with the formation of stable c-TiN and h-AlN wurtzite phases. However, a mix of crystalline (Ti,Al,Si)N and amorphous/nanocrystalline AlN phases can be formed in the nanocomposite coatings [57]. The studies have shown that with the low Al content in the Ti\(_{1-x}\)Al\(_x\)N coating, a solid solution (substitutional solution) of Al forms in the crystalline lattice of TiN, with the cubic structure. At \( 0 \leq x \leq 0.40 \), the films with the NaCl structure were synthesized, while the interplanar distance \( d_{002} \), measured from the X-ray spectra, decreased linearly with an increase of AlN in the film [58]. A significant factor is the irregular distribution of the Al concentration over the volume of the film material [59]. Based on the Al content in the coating under study (about 25 at.%), it is possible to predict hardening due to the formation of a solid solution (Fleischer model) [60].

The selected area electron diffraction (SAED) patterns presented in Figure 9 show that the grain sizes in the wear-resistant coating layer are substantially smaller than their sizes in the transition layer. There is a noticeable grain texture in the transition layer. The interplanar distances correspond well to the interplanar distances of the TiN phase (Fm3m) (2.48, 2.14, 1.51, 1.29, 1.23, and 1.065). The electron diffraction pattern from the wear-resistant layer shows a part of the ring with the interplanar distance of 0.272 nm, earlier associated with the AlN phases (P6.3mc) from the (100) planes.
The atomic number \( z \) of elements contained in each layer. Light layers contain more Ti (\( z = 22 \)), while dark layers contain more Al (\( z = 13 \)). When Al is dissolved in the TiN compound, the interplanar distance \( d_{002} \), measured from the X-ray spectra, decreases monotonically (exponentially) according to the law of X-ray attenuation (\( I = I_0 \exp(-\mu t) \)).

Figure 10. Analysis of the elemental compositions of the transition and wear-resistant layers of the coating is shown in Figure 10. The adhesion of tungsten from the substrate to the transition layer is presented, as well as the content of aluminum and silicon in the wear-resistant layer.

There is almost complete identification of the elemental composition of a wear-resistant layer of all coatings under study based on the system (Ti,Al,Si)N. The average composition in the thickness of a layer is (Ti\(_{0.70}\),Al\(_{0.25}\),Si\(_{0.05}\))N. However, the concentration of Al and Ti in light and dark sub-nanolayers differs. The brightness of a layer during the tests in the z-contrast mode in SEM is determined by the atomic number \( z \) of elements contained in each layer. Light layers contain more Ti (\( z = 22 \)),
while dark layers contain more Al ($z = 13$). When Al is dissolved in the TiN compound, the aluminum atoms replace the titanium atoms in the formation of the $(\text{Ti}_{1-x}\text{Al}_x)\text{N}$ phase, which retains a cubic structure of B1 NaCl type [61,62]. The XRD patterns of the studied coatings (S1–S7) and the substrate (S0) are shown in Figure 11. The substrate contains the main phases of WC and Co. The Co lines are not shown in Figure 11 because of their low intensity. Lines from the substrate are present on all diffraction patterns of samples with coatings. Since the coatings S2–S7 have the same average chemical composition, and the coating thickness increases from S2 to S7, the intensity of the substrate lines monotonically (exponentially) decreases with an increase in thickness according to the law of X-ray attenuation ($I = I_0 \exp(-\mu t)$).

The results of microhardness measurements are presented in Table 3. The minimum, maximum, and average values of microhardness for 10 measurements are given. The Ti–TiN–$(\text{Ti,Al,Si})\text{N}$ (S2–S7) coatings of different thicknesses are characterized by almost equal microhardness.

Based on the hardness measurement data, the obtained data are generally consistent with the earlier hardness measurements of the (Ti,Al,Si)N system based on a coating with the corresponding Ti/Al ratio in the coating composition. There is an insignificant increase in hardness with an increase in coating thickness. This fact contradicts some of the data of [1–4]; however, regarding the nano-composition structure of the coating, during its deposition, no crystal growth and no noticeable change in the mechanical properties are observed.

Figure 11. The XRD patterns of investigated coatings: S1–S7 substrate and coating; S0 substrate (WC–Co).

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Table 3. Results of measurements of microhardness for the coatings under study.

| Sample | Microhardness, HV<sub>0.1</sub> | Min. | Max. | Ave. |
|--------|---------------------------------|------|------|------|
| S1     | 2150                            | 2370 | 2240 |      |
| S2     | 2370                            | 2790 | 2410 |      |
| S3     | 2310                            | 2710 | 2450 |      |
| S4     | 2350                            | 2715 | 2420 |      |
| S5     | 2390                            | 2560 | 2480 |      |
| S6     | 2320                            | 2620 | 2480 |      |
| S7     | 2325                            | 2670 | 2460 |      |

The results of the scratch tests are shown in Figures 12 and 13.

The results of testing of coating S1 (TiN), which showed the values of the critical loads \( L_{c1} = 34 \) N and \( L_{c2} = 37 \) N at a scratch test, were chosen as the base value of the adhesion bond strength. Figure 12 shows that the maximum fracture force for the coating in sample S3 is \( L_{c2} = 47 \) N, and the first cracks appear at \( L_{c1} = 27 \) N. A study of a scratch surface in SEM showed that, when the normal force \( L_{c1} \) is reached, the coating fails under a mechanism of recovery spallation (Figure 14a,b), and when \( L_{c2} \) is reached, the coating fails under a mechanism of gross spallation (Figure 14c,d) [63,64]. Figure 14b shows a crack along the boundary of the coating substrate (indicated by an arrow); however, no delamination of coating occurred.

On samples S4, S6, and S7, spallation of the coating under \( L_{c2} \) occurs as compressive spallation/gross spallation (Figure 14c,d). In this case, a coating spalls over a wide surface in front of an indenter, which indicates the effect of tensile stresses on the coating–substrate interface. The width of spallation exceeds the width of the scratch by 3–5 times. It has been found that, in coatings S4, S6, and S7, no cracks or partial spallation of coatings occur during scratching (i.e., there is no \( L_{c1} \)), and the coating spalls entirely at \( L_{c2} \). Figure 14c shows that, on sample S4, the coating fails with complete delamination from the substrate (i.e., on the boundary of the interface). On samples S6 and S7, the process of coating failure proceeds with the formation of extended cracks, causing delaminating of the coating parallel to the substrate surface and destruction over a large area. The SEM image (Figure 14d) shows that...
the failure surface passes through different TiN–(Ti,Al,Si)N sublayers (lighter and darker areas are indicated by arrows). Meanwhile, no spallation of the TiN sublayer except for local zones occurs, which indicates a good adhesion of the sublayer to the substrate. As a result, under actual cutting conditions, after the failure of the wear-resistant layer, a tool continues its operation due to the presence of a sublayer remaining on the substrate. In this case, it can be assumed that the tool operating time after spallation of a wear-resistant layer is comparable or slightly shorter than the operating time of a tool with a monolayer TiN coating.

Figure 13. Scratch tests of the coatings under study.

Figure 14. Wear pattern on coatings in scratch testing: (a) S2, (b) S4, (c) S6, and (d) S7.
The normal load \( L_{c2} \) for coatings with a wear-resistant layer thickness exceeding 5790 nm is lower than for a coating with a wear-resistant layer thickness of 3670 nm, which could possibly be related to the presence of residual stresses in the coating, which increase with an increase in the wear-resistant layer thickness. The mechanisms of failure of all coatings considered in this paper are brittle types, which is explained by the high hardness of the coating and substrate [63]; however, for the same reason, it is difficult to analyze the types of failure from the acoustic emission graphs.

### 3.2. Study of Cutting Properties of Tools with Coatings under Study

Figure 15 shows the relationships between the tool life of a cutting tool and the thickness of a coating wear-resistant layer for longitudinal turning of steel C45. An obvious trend is that, when the cutting speed is increased, the tool life of tools with thicker coatings (especially S6 and S7) decreases compared with a tool with a thinner coating. At a cutting speed of 350 m/min, a tool with coating S7 shows a tool life that is almost identical to that of an uncoated tool. This tendency can be explained by the increase in temperature in the cutting zone, which results in more intensive wear and the failure of thick coatings with a high level of internal residual stresses. A tool with the S4 coating shows the longest relative tool life at all cutting speeds. A tool with coating S3 shows the longest relative tool life with an increase in cutting speed.

![Graphs showing tool life vs. thickness of wear-resistant coating layer](image)

**Figure 15.** Relationship between the tool life of a carbide tool and the thickness of a wear-resistant coating layer during longitudinal turning of steel C45 \((f = 0.25 \text{ mm/rev}, a_p = 1 \text{ mm}, \text{ and } (a) v_c = 250 \text{ m/min}, (b) v_c = 300 \text{ m/min} \text{ (c), } v_c = 350 \text{ m/min}).**

We consider the relationship between the wear resistance of cutting tools with the coatings under study and the cutting speed (Figure 16). The curves clearly show that tools with coatings with large thicknesses of wear-resistant layers (S6 and S7) show a period of resistance that is significantly reduced with an increase in the cutting speed. This decrease occurs much more actively in comparison not only...
with tools with coatings S2–S5, but also with a tool with TiN coating and an uncoated tool. The least active decrease of wear resistance is observed for tools with coatings S2–S4.

Figure 15. Relationship between the tool life of a carbide tool and the thickness of a wear-resistant coating layer during longitudinal turning of steel C45 ($f = 0.25 \text{ mm/rev}$, $a_p = 1 \text{ mm}$, and $v_c = 250, 300, \text{ and } 350 \text{ m/min}$).

The studies carried out earlier in turning cryogenic steel AISI 321 ($v_c = 85–150 \text{ m/min}$) using the tools with the Ti–TiN–(Ti,Al,Cr)N nano-structured coatings found that the longest tool life was shown by the tools with the wear-resistant layer thicknesses of 5 and 3 μm [65]. It should be noted that steel AISI 321 is a hard-to-cut material (ISO 513 Material Group M), while steel C45 under study in the article is included in ISO 513 Material Group P. Thus, it can be predicted that for machining materials from ISO 513 Material Group M, it is reasonable to use coatings with wear-resistant layer thicknesses of 3–5 μm, while for machining materials from ISO 513 Material Group P, the reasonable wear-resistant layer thicknesses are 5–7 μm.

3.3. Study of Coating Wear and Failure on Cross Section

The mechanism of wear and failure of the tool with TiN coating is well studied. In general, brittle fracture occurs with the formation of thorough transverse cracks and branching-type cracks. Similar mechanisms of failure can be seen in Figure 17. There are no boundaries of layers in the monolithic structure of TiN, which are capable of inhibiting or transforming the development of cracks.

The cutting tool with nano-structured coatings shows the mechanism of failure, which has several principal differences from the one observed for monolithic coatings. The main difference is the ability of a sub-nanolayer structure to inhibit the development of cracks. Meanwhile, as shown earlier [29], the mechanism of failure for the cutting tool with nano-structured coatings with high hardness and low toughness (in particular, (Al,Ti)N coatings with a high content of aluminum) is close to the mechanism of failure for the cutting tool with solid monolithic coatings; that is, such a coating fails as a whole. Meanwhile, the cutting tool with coatings with higher toughness and impact toughness values (in particular, the (Ti,Al,Si)N coatings under study with low aluminum content and the coatings with high zirconium content considered earlier [29]) demonstrate the mechanism of failure and cracking, which is characterized by the inhibition of cracks on the boundaries of the nanolayers.
In particular, the cutting tool with coating S2, the thinnest coating under study, demonstrates the mechanism of cracking shown in Figure 18. The transverse cracks (separated by a short red-dotted line) are transformed into longitudinal cracks and delaminations at the boundaries of sub-nanolayers (marked with a short yellow-dotted line). The boundaries of the sub-nanolayer are indicated by a long green-dashed line. This process restrains the failure of the coating to a certain extent, allowing it to retain its performance for a longer time.

Figure 17. Mechanism of wear and failure for a cutting tool with coating S1 (Ti–TiN). Turning of steel C45 at cutting speed \( v_c = 250 \text{ m/min} \) (\( f = 0.25 \text{ mm/rev} \) and \( a_p = 1 \text{ mm} \)).

Figure 18. Mechanism of wear and failure for the cutting tool with coating S2. Area adjacent to wear crater on the rake face. Turning of steel C45 during cutting speed \( v_c = 250 \text{ m/min} \) (\( f = 0.25 \text{ mm/rev} \) and \( a_p = 1 \text{ mm} \)). General view of the transverse section (a) and the nature of the destruction of nanolayers (b).

For the cutting tool with coating S3, the presence of extended branching of longitudinal cracks is typical. In particular, Figure 19a shows how a similar crack, which is a combination of local delaminations between sub-nanolayers and ruptures of sub-nanolayers, is developing in the coating structure. It is important to note that this crack, when developing in the coating structure, crosses the boundary of TiN and (Ti,Al,Si)N layers and reaches the substrate. Meanwhile, the crack under study does not cut the structure of the coating completely, and the superficial layers of the coating remain intact.
Figure 18. Mechanism of wear and failure for the cutting tool with coating S2. Area adjacent to wear crater on the rake face. Turning of steel C45 during cutting speed \( v_c = 250 \text{ m/min} \) \( (f = 0.25 \text{ mm/rev} \text{ and } a_p = 1 \text{ mm}) \). General view of the transverse section \( (a) \) and the nature of the destruction of nanolayers \( (b) \).

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Figure 19. Mechanism of wear and failure for cutting tool with coating S3 \( (a) \) and S4 \( (b,c) \).

Thus, since a tool with coating S4 shows the best results under all cutting modes, we consider in detail the nature of failure for this coating. It is important to note that the nature of wear and failure for this coating around the rake face directly adjacent to the cutting edge is balanced without brittle fracture and noticeable cracking (Figure 19b). There is an extensive pickup of the material being machined on the surface of a tool with this coating in the area under consideration.

We consider the nature of crack formation in different areas of coating S4 (Figure 19c). Transverse cracks are formed less often (in particular, in the zone of an embedded microdroplet, and as a rule, they are not thorough). In general, it should be noted that the cracking primarily affects the surface layers of a coating, while the lower layers retain their integrity and, therefore, their performance.

In coatings of higher thickness, starting from coating S5, distinguishing characteristics appear that are not typical for thinner coatings (S2–S4). In particular, as a result of higher internal stresses, the formation of sequences of local delaminations is observed, which is usually a consequence of micro-geometric deviations of the substrate surface (cavities and convexities) or microdroplets embedded in the lower layers of the coating (Figure 20a).

A somewhat different effect on crack formation is caused by defects (primarily microdroplets) embedded in the structure of the coating in the middle part or in the surface layers (Figure 20b). In this case, a microdroplet embedded in the coating structure results in the formation of a closed enveloping crack, from which longitudinal and transverse cracks develop. It is clear that such a network of cracks leads to the failure of this section of the coating and, correspondingly, to a decrease in the performance of the coated tool. It can be argued that this microdroplet is a stress concentrator that adversely affects the hardness and wear resistance of coatings.
We consider the nature of the failure of the cutting tool with coating S6. In the area of the rake face immediately adjacent to the cutting edge, several different mechanisms of failure are observed at once (Figure 20c). Directly on the cutting edge, a fairly balanced wear pattern is observed. Meanwhile, in the area slightly farther from the cutting edge, Figure 20c shows the formation of thorough transverse cracks, with deep penetration (charging) of the material being machined (crack A) and the formation of a network of longitudinal cracks and delaminations, combined with short transverse cracks. In this area, there is significant destruction of the surface layers of the coating, while the middle and inner layers retain their efficiency.

Like the earlier studied cutting tool with coating S5, coating S6 shows the formation of a sequence of delaminations initiated by the defect of the deep layers. In particular, Figure 21 shows such a sequence, initially associated with microroughness (protrusion) on the surface of the substrate.
In this case, it is worth noting a fairly good periodicity of repeating delaminations (through 18–20 sub-nanolayers), which can be associated with cycles of increase in longitudinal compressive stresses and their “dumping” due to the occurrence of delaminations.

**Figure 21.** Mechanism of wear and failure for the cutting tool with coating S7: General view of the cross section at the border of the wear crater (a); Area A: Destruction of the coating at the boundary of the wear crater (b); (c) Area B: Microdroplets embedded in the structure of the coating; (d) Area C: Crack propagation is inhibited by adhesion bridges between sub-nanolayers (denoted by a yellow-dotted line, while arrows indicate internal tension stresses).
Coating S7, the thickest of the coatings under study, demonstrates the mechanism of brittle fracture with the formation of a network of longitudinal and transverse cracks (Figure 21). Meanwhile, the coating is characterized by sufficient plasticity, since delamination and longitudinal cracks dominate (Figure 21, Area A). Meanwhile, there are transverse cracks that cut through the structure of the nanolayers. A confirmation of the sufficient plasticity of coating S7 is the formation of bond bridges between the nanolayers, which inhibit the crack propagation (Figure 21, Area C).

The boundaries of nanolayers are able to inhibit the crack propagation by transforming transverse cracks into delaminations at the boundaries of the sub-nanolayers, which is less dangerous in terms of coating failure. Figure 21 Area C shows how sub-nanolayer III is deformed plastically but does not fail in the crack propagation zone. Meanwhile, sub-nanolayer II is also plastically deformed by forming a thinner ‘neck’ in the zone of intersection of the crack. Sub-nanolayer I is completely ruptured in the crack propagation zone. In this zone, sub-nanolayer IV is not damaged. When reaching the boundary of sub-nanolayers III and IV, the crack changes direction, transforming into an interlayer delamination. Area B (Figure 21) presents a picture of the internal defects of the coating delaminations and transverse cracks initiated by a microdroplet embedded in the coating structure at the early stage of deposition of the coating.

Here, there is a kind of “heredity” of the defect in the form of delamination, repeated through a certain number of sub-nanolayers. Transverse cracks are formed in the center and in the edge zones of this area. However, this defect remains undeveloped; that is, it does not lead to direct coating failure. A model for formation of these defects in the coating nano-structure is discussed above in more detail.

4. Conclusions

This study investigated the properties of Ti–TiN–(Ti,Al,Si)N multilayer composite nano-structured coatings with the thicknesses of its wear-resistant layers of 2.0, 3.5, 5.0, 7.0, 11.0, and 15.0 µm. It is found that the coatings under study are characterized by close values of microhardness (HV0.1 of about 24 GPa). Meanwhile, the strength of the adhesive bond with the substrate LC2 is within a range of 34–47 N and does not correlate directly with the tool life during cutting tests. The cutting tests carried out in longitudinal tuning of steel 45 (at vc =250, 300, and 350 m/min) show that the longest tool life was shown by the tools with the wear-resistant layer thicknesses of 5–7 µm (and, respectively, the total coating thicknesses of 6–8 µm). These values are slightly higher that the thicknesses of 3–5 µm usually used by tool manufacturers. Moreover, the tests carried out earlier in turning steel AISI 321 (ISO 513 Material Group M) using the tools with the coatings of various thicknesses found that the longest tool life was provided by a coating with the wear-resistant layer thickness of 3–5 µm [65]. Thus, the optimal thickness of a wear-resistant layer of the coating depends on machining conditions and the material group to which the material being machined belongs.

The study of the relationship between the tool life of a coated tool and the cutting speed shows that a tool with a thicker wear-resistant layer (11 and 15 µm) tends to drastically reduce its tool life with an increase in cutting speed. Meanwhile, a tool with a smaller thickness of wear-resistant layer (2–5 µm) shows a significantly smaller decrease in wear resistance with an increase in cutting speed.

The studies of the process of coated tool wear and failure on the cross section show a marked difference in these processes for thin and thick coatings. For coatings with a thicker wear-resistant layer, formation of a sequence of internal delaminations is typical and usually associated with deep defects, such as embedded microdroplets, and surface defects of the substrate. Meanwhile, thicker coatings show the effect of amplification associated with the internal defects of microcracks as the coating thickness increases.

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