Comparative Analysis of Tribological and Functional Properties of Multilayer Composite Nanostructured Coatings Based on Nitrides of Cr, Mo, Zr, Nb, and Al

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Abstract. The article describes the results of the studies focused on the properties of multilayer composite coatings with nanolayer structures based on nitrides of Cr, Mo, Zr, Nb, and Al. Tests were carried out to study the influence of temperature varying in the range of 600–1000 °C on the value of the adhesion component of the coefficient of friction (COF). The investigation was also focused on the cutting properties of carbide tools with the coating understudy during the turning of AISI 321 steel at various cutting speeds. The study revealed the relationship between the value of the adhesion component of the COF and the tool life of coated cutting tools. The tool with the Cr,Mo-(Cr,Mo)N-(Cr,Mo,Al)N coating under study demonstrated good wear resistance on the tool's rake face during the cutting of corrosion and heat-resistant AISI 321 steel. With an increase in the cutting speed, the cutting properties of the tool with the coating understudy increase compared to the tools with other coatings or uncoated tools.

Keywords: Physical coatings · Coefficient of friction · Tool life · Thermo stability · Tool wear

1 Introduction

Tribological and frictional properties of coatings are interrelated and significantly influence the performance properties of products on the working efficiency of cutting tools. It should be considered that with an increase in a temperature to 500–1000 °C, which is typical for the cutting zone, the tribological properties of the coatings change significantly. This change relates both to an increase in the material ductility and the formation of tribologically active oxide compounds (Magneli phases) on the surfaces of the coatings. Certain metal oxides are characterized by particularly favorable tribological properties at high temperatures in combination with high thermostability. The inclusion of such metals or their nitrides in the compositions of the coatings for cutting tools contributes to an increase in their working efficiency.

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2 Literature Review

At high temperatures, nitrogen is replaced by more active oxygen, and thus, oxides are formed instead of nitrides [1]. Aluminum oxide Al_2O_3 is characterized by favorable properties [2, 3]. During the heating of samples with the Al2O3-based coatings, the COF increases to a certain temperature (about 700 °C) and then the COF decreases significantly [4, 5]. During heating of the coatings based on the nitrides of such metals as Al-doped Ti, Cr [6], V, Nb, Mo, W [7], and Y [8], the oxide phase of Al_2O_3 is being formed, sometimes in the form of a continuous surface film of nanometric thickness [6–8]. Apart from aluminum, molybdenum [9, 10] (which forms MoO_3 oxide [11, 12]) and chromium (which forms Cr_2O_3 oxide) [13] are also able to form strong tribologically active oxide films. Similar films can also be formed at temperatures above 700 °C [12]. However, during heating to temperatures above 900 °C, the coatings with high molybdenum content and chromium begin to lose their performance properties due to the too active oxidation process [14].

Thus, it may be noted that the formation of oxides in the surface layers of the coatings may have both favorable (due to the improvement of tribological parameters and formation of protective films) and unfavorable (due to the total fracture of the surface structure of the coating) influence on the performance properties of the coatings.

The studies focused on the tribological properties find that the coatings with multilayer structures demonstrate better values of the COF, hardness, and adhesion to the substrate compared to the coatings with monolayer structures [15, 16]. However, the COF does not always play a crucial role in the cutting properties of coated tools. In particular, although for the (Ti,Al)N coatings, the value of COF is slightly higher than for the (Cr,Al)N coating, a tool with the (Cr,Al)N coating demonstrated higher wear resistance during turning [17]. The noticeable influence on the cutting process is exercised by forming oxides of Cr_2O_3 and especially MoO_3, which contribute to a noticeable decrease in the COF at temperatures typical for the cutting zone [18]. With an increase in temperature, the hardness of most coatings decreases, but in some cases, the opposite effect can occur. In particular, as the temperature rises to 800 °C, the coherency strains are being formed in the (Ti,Al,Mo)N coating, and they prevent the sliding of dislocations and thus improve the hardness of the coating [19, 20]. The COF of the coatings first increases as the temperature rises to 500–600 °C and then decreases with further growth of the temperature [21, 22]. The development of coatings with a multi-component composition of a wear-resistant layer (including nitrides of three and more metals) contributed to an additional mechanical and performance properties improvement compared to the coatings with mono- or two-component composition [21, 23]. The main challenge of the article is to study the influence of chromium and molybdenum on the tribological properties of the coatings. The multilayer composite coatings based on nitrides of Cr, Mo, Zr, Nb, and Al were chosen as the main object of the study.

3 Research Methodology

The coatings under study were deposited using the filtered cathodic vacuum arc deposition (FCVAD) technology [16, 24, 25]. The technology was implemented on the VIT-2 unit (developed by VIT–IDTI RAS), which included two evaporators with a pulsating magnetic field and an evaporator with a curved plasma duct longitudinal, continuous, and uniform magnetic field [26, 27].

It is extremely difficult, if possible, to measure the COF directly during the cutting of materials. On the other hand, the standard methods for measuring the COF do not consider the essential features of the cutting process since they do not consider the temperature in the cutting zone and related factors (for example, oxidation and diffusion processes). Thus, it is reasonable to use a measurement method that, on the one hand, simulates the cutting conditions efficiently and, on the other hand, makes it possible to measure the COF adequately and accurately. The current study used the equipment and method described in detail in [21, 22].

In the presented mathematical model, a spherical indenter, which is made of the material identical to the tool material and which simulates a contact patch between a cutting tool and a workpiece being machined, rotates around its own axis, being compressed by two plain parallel inserts made of the material identical to the material being machined [28]. The inserts compress the indenter with certain forces, and a certain temperature in the range from zero to 1000 °C is provided in the entire system. The force F_{exp} required to rotate the indenter compressed by the inserts is measured. The force relates to the shear strength τ_n of adhesive bonds.

The strength τ_{nn} of adhesion bonds on the cut is determined from the relation [29]:

$$\tau_n = \frac{3}{4}\frac{F_{exp}}{\pi}\frac{R_{exp}}{r_{ind}^3} \tag{1}$$

where F_{exp} is the circumferential force on the disk, rotating the indenter; R_{exp} is the radius of the disk in which the indenter is fixed; and r_{ind} is the radius of the indent on the samples.

Normal stresses are defined as follows [29]:

$$p_n = \frac{N}{\pi r_{ind}^2} \tag{2}$$

Thus, the adhesion (molecular) component of the COF is defined as follows [29]:

$$f_M = \frac{\tau_n}{p_n} = \frac{3}{4}\frac{F_{exp}}{N}\frac{R_{exp}}{r_{ind}} \tag{3}$$

where p_n is normal stresses acting on the surface of the sphere.

The hardness (HV) of the coatings was determined by measuring the indentation at low loads according to the method proposed by Oliver and Pharr, which was conducted on a micro-indentometer hardness tester (CSM Instruments) at a fixed load of 10 mN. SNUNISO 1832:2012 carbide (WC+15% TiC+6% Co) inserts were used to study the cutting properties of coated tools. Indenters of similar compositions (WC+15% TiC +6% Co) were chosen to investigate the tribological properties of the coated samples. An ACU 500 MRD lathe (Sliven) with a ZMMCU500MRD variable-speed drive was involved in the investigation focused on the cutting properties of the tools with the coatings under study. The cutting geometry parameters were: $\gamma = -7°$, $\alpha = 7°$, $\lambda = 0$, r = 0.4 mm, at the following cutting conditions: $f = 0.1$ rpm, $a_p = 0.5$ mm, and $v_c =$ 60, 80, 100, and 120 m/min. The nanostructure of the coating was examined with a JEM 2100 (JEOL, Japan) transmission electron microscope (TEM) using EDX system INCA Energy (OXFORD Instruments) to determine the chemical composition of the coatings.

4 Results

The results of the comparative analysis on the chemical composition of the coatings under study are presented in Table 1. All the coatings are characterized by low content of aluminum (within 6–16 at%). Since there may be a gradient change in the scope of elements in the coating nanolayers, Table 1 also indicates ranges of such change.

Table 1. Chemical composition of the coatings under comparison, at %.

Coating composition	Cr	Mo	Al	Ti	Zr	Nb	Si
Cr,Mo-(Cr,Mo)N-(Cr, Mo,Al)N	67–77	18–25	6–13	–	–	–	–
Ti-TiN-(Ti,Cr,Al,Si)N	32–44	–	6–12	44–53	–	–	2–3
Zr-ZrN-(Zr,Al,Si)N	–	–	6–11	–	87–91	–	2–3
Zr-ZrN-(Nb,Zr,Cr,Al)N	25–33	–	6–12	–	25–35	25–35	–
Zr,Nb-(Zr,Nb)N-(Zr,Nb, Al)N	–	–	7–16	–	39–43	39–45	–
Cr,Mo-(Cr,Mo,Zr,Nb)N-(Cr,Mo,Zr,Nb,Al)N	43–50	16–23	6–8	–	11–14	12–14	–
Ti-TiN-(Ti,Al,Cr)N	29–38	–	8–6	45–55	–	–	–

The nanostructures of the coatings under study are considered (Fig. 1). The presented images depict the nanolayer structures of all the coatings under investigation. The first six coatings have close values of the nanolayer period λ (100–200 nm), and two options – with an extremely large (302 nm) and an extremely small (10 nm) nanolayer period λ – are considered for the Ti-TiN-(Ti,Al,Cr)N coatings. The above methods may be applied to study both the influence of the chemical composition of the coatings and the impact of their nanostructure on the value of the adhesion component

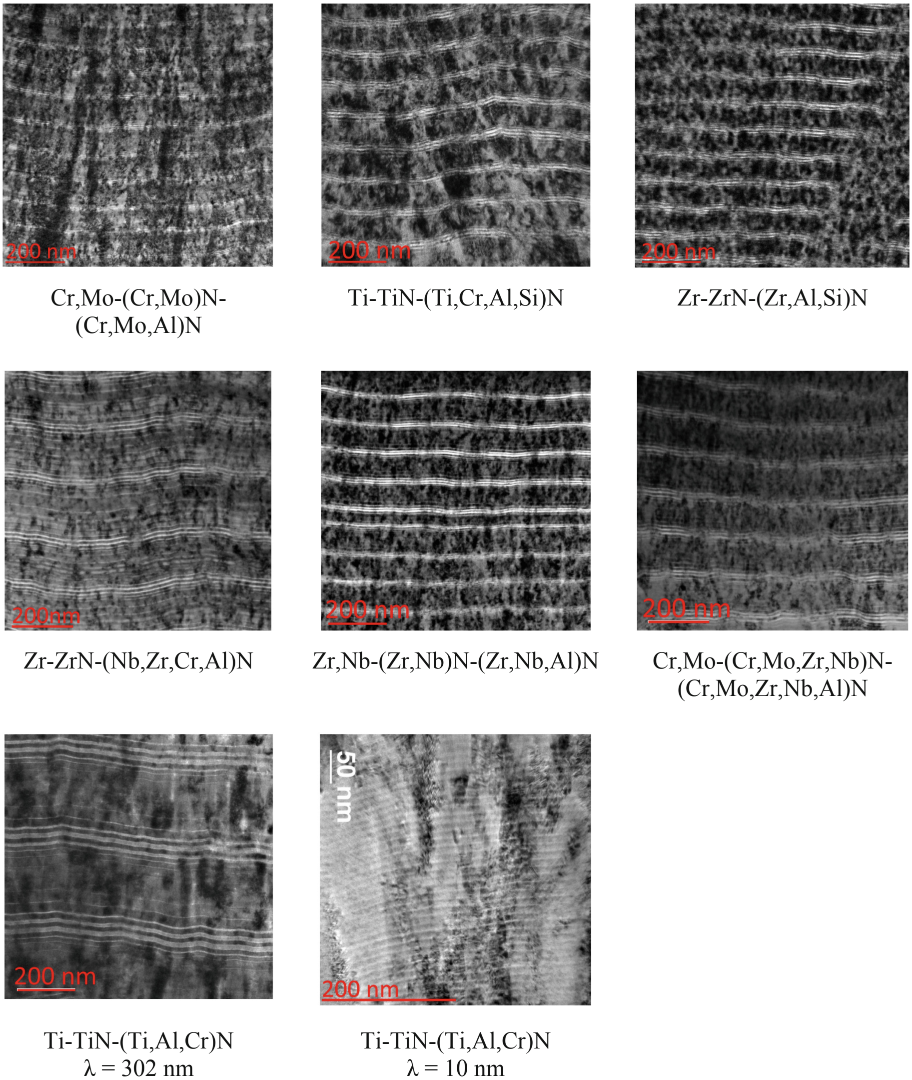

Fig. 1. Nanostructure of the coatings under study.

of the COF. Table 2 gives the results of the measurements of the hardness HV and the nanolayer period λ of the coatings under study. The value of λ has no significant influence on hardness [34, 35]. The tribological properties (the value of f_{adh}, the adhesion component of the COF) of the samples under study are compared (Fig. 2). While at the temperature of 500 °C, the value of f_{adh} for the sample with the Cr,Mo-(Cr,Mo)N-(Cr,Mo,Al)N coatings is slightly higher compared to the values of f_{adh} for the samples with the coatings based on ZrN (Zr-ZrN-(Zr,Al,Si)N and Zr-ZrN-(Nb,Zr,Cr,Al)N) and is close to the values of f_{adh} for the samples with the coatings of

Ti-TiN-(Ti,Cr,Al,Si)N; Zr,Nb-(Zr,Nb)N-(Zr,Nb,Al)N and uncoated samples, then at the temperature of 800 °C, the sample with the Cr,Mo-(Cr,Mo)N-(Cr,Mo,Al)N coating demonstrates the lowest value of f_{adh} among all the samples under study. With a further increase in the temperature to 900 and 1000 °C, the tendency intensifies and the importance of f_{adh} for the sample with the Cr,Mo-(Cr,Mo)N-(Cr,Mo,Al)N coating is noticeably lower than for the other samples under study. At the given temperatures, the values of f_{adh} for all samples with the (Ti,Cr,Al)N-based coatings become even higher than for the uncoated sample.

Thus, the value of f_{adh} increases upon heating from 500 to 800 °C for all the samples under study. During heating to 900 and 1000 °C, the value of f_{adh} begins to decrease for most of the coated samples under study but almost does not change for the uncoated sample and the samples with the coatings containing Nb and not containing Mo: (Zr,Nb-(Zr,Nb)N-(Zr,Nb,Al)N and Zr-ZrN-(Nb,Zr,Cr,Al)N). The Cr,Mo-(Cr,Mo, Zr,Nb)N-(Cr,Mo,Zr,Nb,Al)N coating, containing both Mo and Nb, demonstrates a slight decrease in f_{adh} at the given temperatures. Based on the above, it can be assumed that the presence of Mo in the composition of the coating contributes to a decrease in f_{adh} upon heating to a temperature of 900 °C and above, while the presence of Nb, on the contrary, inhibits such a decrease. This phenomenon can be explained: at high temperatures, the coatings based on the nitrides of Mo, Cr, and Al form solid and dense oxide films of MoO_3, Cr_2O_3, $(Cr,Al)_2O_3$, and Al_2O_3 [32] that contribute to a decrease in the value of f_{adh}. At the same time, the Nb-containing coatings form soft porous oxides of Nb_2O_5 and $CrNbO_4$ [33] that inhibit a decrease in f_{adh}. Zirconium oxide ZrO_2 is also a solid and dense compound. It can positively influence the tribological properties of the coatings, which may explain a decrease in f_{adh} for the ZrN-based coatings.

The influence of various coatings on the flank wear rate of carbide cutting tools is considered. Figure 3 depicts the relationship between $\boldsymbol{H_m}$ (the specific value of the flank wear rate per one meter of the cutting path) and the cutting speed during turning with carbide tools with the coatings under study. The lower rake wear rate at all cutting speeds was demonstrated by the tool with the Ti-TiN-(Ti,Al,Cr)N coating (λ = 10 nm). The tool with the Cr,Mo-(Cr,Mo)N-(Cr,Mo,Al)N coating understudy also showed good resistance to wear on the rake face. It should be noted that with an increase in the cutting speed, the difference in the wear rate on the samples with the Ti-TiN-(Ti,Al,Cr) N (λ = 10 nm) and Cr,Mo-(Cr,Mo)N-(Cr,Mo,Al)N coatings slightly reduces. Such an effect may relate to the fact that the temperature rises with an increase in the cutting speed and, accordingly, it intensifies the formation of the MoO_3 oxide film, which has a positive influence on the cutting conditions. In the course of the earlier tests, the tool with the Cr,Mo-(Cr,Mo,Zr,Nb)N-(Cr,Mo,Zr,Nb,Al)N coating showed the highest wear resistance in turning of AISI 1045 steel. Meanwhile, in the course of a current series of cutting tests, its wear resistance appeared to be lower compared to the tools with the Ti-TiN-(Ti,Al,Cr)N (λ = 10 nm) and Cr,Mo-(Cr,Mo)N-(Cr,Mo,Al)N coatings. Since corrosion- and heat-resistant AISI 321 steel (HB 179 MPa) is a hard-to-cut material (M20-M25 ISO), the cutting conditions for AISI 321 steel differ significantly from the cutting conditions for AISI 1045 steel [34]. The steel being machined contains Cr (18 at %) and Ni (12 at%), which significantly influences the nature of its machining and wear of the cutting tool.

Table 2. Results of coating hardness measurements (HV, GPa).

Coating	HV, GPa	λ, nm
Cr,Mo-(Cr,Mo)N-(Cr,Mo,Al)N	24.0 ± 1.6	82
Ti-TiN-(Ti,Cr,Al,Si)N	36 ± 1	98
Zr-ZrN-(Zr,Al,Si)N	28 ± 1	110
Zr-ZrN-(Nb,Zr,Cr,Al)N	26 ± 1	205
Zr,Nb-(Zr,Nb)N-(Zr,Nb,Al)N	23.2 ± 3.6	96
Cr,Mo-(Cr,Mo,Zr,Nb)N-(Cr,Mo,Zr,Nb,Al)N	28.2 ± 2.1	138
Ti-TiN-(Ti,Al,Cr)N (λ = 302 nm)	29.82 ± 1	302
Ti-TiN-(Ti,Al,Cr)N (λ = 10 nm)	30.64 ± 1	10

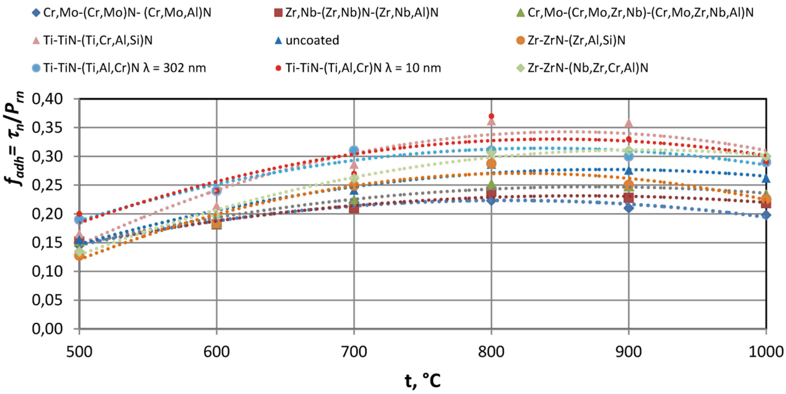

Fig. 2. Comparison of the tribological properties (the value of fadh, the adhesion component of the COF) for the samples under study.

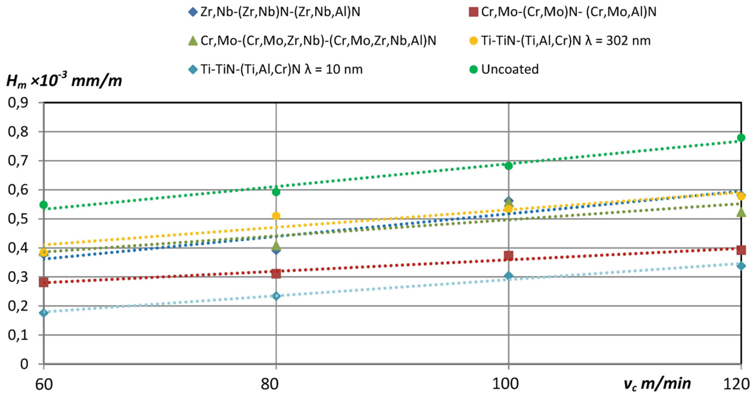

Fig. 3. Relationship between Hm (the specific value of the flank wear rate per one meter of the cutting path) and the cutting speed during turning with carbide tools with the coatings under study.

5 Conclusions

The properties of multilayer composite coatings of various compositions with a nanostructured wear-resistant layer were studied. The conducted studies have found the following.

The Cr,Mo-(Cr,Mo)N-(Cr,Mo,Al)N coating under study demonstrated the lowest value of f_{adh}, the adhesion component of the COF, among all the tested samples, over the entire temperature range of the study.

The presence of Mo in the composition of the coating contributes to a decrease in f_{adh} upon heating to a temperature of 900 °C and above, while the presence of Nb, on the contrary, inhibits such a decrease in f_{adh}.

The tool with the Cr,Mo-(Cr,Mo)N-(Cr,Mo,Al)N coating under study demonstrated good wear resistance on the tool's rake face during the cutting of corrosion and heat-resistant AISI 321 steel. With an increase in the cutting speed, the cutting properties of the tool with the coating understudy increase compared to the tools with other coatings or uncoated tools.

Further planned research on this topic is to study the effect of the magnitude of the nanolayer period λ on the properties of various compositions' coatings and the cutting properties of tools with such coatings.

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