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# Tribotechnical characteristics of nanostructured multilayer composite coatings on tools for machining heat-resistant alloys

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The paper presents the results of theoretical and experimental research of tribotechnical characteristics (tool wear on the back surface, tool durability period, critical cutting path length before blunting) of innovative nanostructured multilayer composite coatings on the tool for milling heat-resistant alloys of aerospace parts.

**Keywords:** nanostructured composite multilayer coatings, heat resistant alloys, wear resistance, tool durability period, cutting path length.

## 1. Introduction

Modern machine-building production urgently needs highly efficient tooling to ensure the introduction and machining of innovative advanced types of structural materials with unique physical, mechanical and thermal properties, in particular, parts of the power part of gas turbine engines while ensuring high requirements for the quality indicators of machined surfaces with the highest productivity at the lowest cost [3, 4]. It is also known [5–10] that the operational properties of cutting tools to a greater extent predetermine the level of technical capabilities, the need for development and application of modern expensive high-performance metal-cutting equipment with numerical program and adaptive control. The analysis of domestic and foreign publications [7, 8] has shown that at high temperature-force loads on the cutting wedge during mechanical processing of heat-resistant steels and alloys to improve wear resistance the most effective are nanostructured coatings obtained by physical vapor deposition (PVD) methods. The process of metal processing by cutting is a complex process by its physical essence [2, 10–16]. When a cutting tool is in contact with the machined material, increased pressures (up to 2000 MPa and more) are manifested, which lead to an increase in temperature (up to 1000...1300°C) and contribute to the appearance of high strain rates (up to  $10^6 \text{ s}^{-1}$ ) etc. This process leads to the formation of favorable conditions for adhesive bonding of contacting materials with their subsequent detachment and wear, mutual diffusion and surface vaporization, changes in the structural-phase composition and generation of thermo-electromotive force. The above conditions have a significant impact on the properties and state of the contact surfaces of the cutting tool and workpiece material. During implementation of the cutting machining process, the main source of increased temperature (heat) and factors of formation of near-surface layers is friction. Friction occurs under conditions of high

temperatures at juvenile contact, accompanied by plastic deformation, therefore, the adhesive (molecular) component prevails in the contact processes [11–18]. Despite the development of high-performance types of tool materials and various ways to improve their cutting properties, one of the most promising ways to improve the milling of hard-to-machine materials is the formation of modified multilayer, multicomponent, nanostructured composite wear-resistant coatings on their working surfaces [10–18].

The purpose of this work is to conduct tribotechnical studies of end mills with nanostructured coatings for machining heat-resistant chromium-nickel alloys.

## 2. Theoretical and experimental background

To analyze and explain the contact processes during metal cutting, the stress-strain state of the cutting wedge under various temperature and force conditions, various approaches, and methods of describing these phenomena are used. In addition, to date, the structural-energetic description of the process of contact interaction between the machined material and the surfaces of the cutting tool is predominantly used and developed. At the same time, the cutting process is considered as an open thermodynamic system with dissipation of energy spent on any process, i. e., the dissipative function (energy spent per unit time per unit area) of external forces is spent by plastic deformation of machined material, changes in the shape (wear) of cutting tool and structural-phase transformations of surface layers of machined and tool materials [2, 5]. At the same time, according to the studies [19–25], when developing coatings with an innovative concept: multilayer composite architecture with nanoscale structure and alternating layers of nanoscale thickness of different composite composition and functional purpose for a new generation of cutting tools, it is necessary to consider the specific influence of temperature and force conditions of cutting to adapt the friction surfaces. Thus,

one of the directions of improvement of physically deposited coatings is the creation of combinations of compositions allowing to use materials with lubricating and protective properties in the conditions of high-temperature processing due to the formation of secondary structures in the form of oxides of titanium, aluminum, chromium, etc.

### 3. Equipment and methods of experimental research

At the preliminary stage to reduce the time to determine the effective composition of coatings and their application modes, as well as the costs of both tooling and cathode material, a number of adhesion tests were carried out. To estimate tribotechnical parameters ( $\tau_{mn}$ ,  $p_{rn}$ ,  $\tau_{mn}/p_{rn}$ ), an experimental method was used [11]. This method is based on the physical model (Fig. 1) and the developed installation (Fig. 2) [12], which in the first approximation reflects the real conditions of friction and wear at local contact. According to this model, a spherical indenter (2) in Fig. 1, made of H10F tool material with different coatings (simulating one irregular rubbing-solid contact) compressed by two plane-parallel specimens (1) made of chrome-nickel alloys — EP99; EK61 (with high accuracy and cleanness of contact surfaces) rotates under the load  $N$  around its own axis. The force  $F_{ex}$  consumed to rotate the indenter and applied to the cable (4) placed in the disc slot (3) is mainly related to the shear strength  $\tau_{mn}$  of the adhesive bonds. The other end of the cable is connected to the elastic elements (6,9) and the recording device (8), by means of which the driving forces for the rotation of the indenter (7) are recorded. Figure 2 shows a diagram of the adhesion testing setup, with a spherical indenter made of instrumental material (1) clamped between two flat-parallel specimens (10) with a disk (2), which is driven through a cable (4) by a pulling mechanism (7), with elastic elements (6,9) and a recording device (8) in the figure.

Ensuring the temperature regime in the contact zone of the indenter with the samples of the processed material in a wide range of variations (0–1300°C) uses the electrical contact method, high voltage is supplied from the power-controlled transformer (14) to the terminals (11) isolated from the body [11].

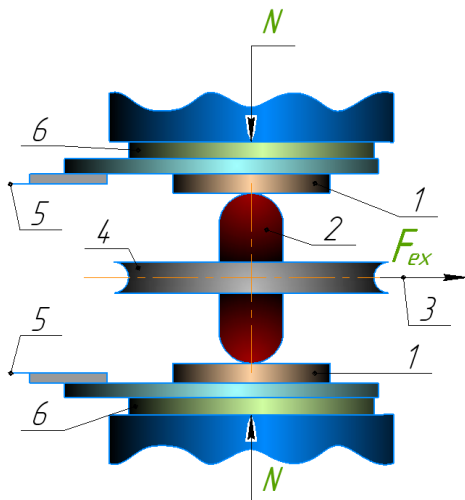


Fig. 1. (Color online) Friction model contact.

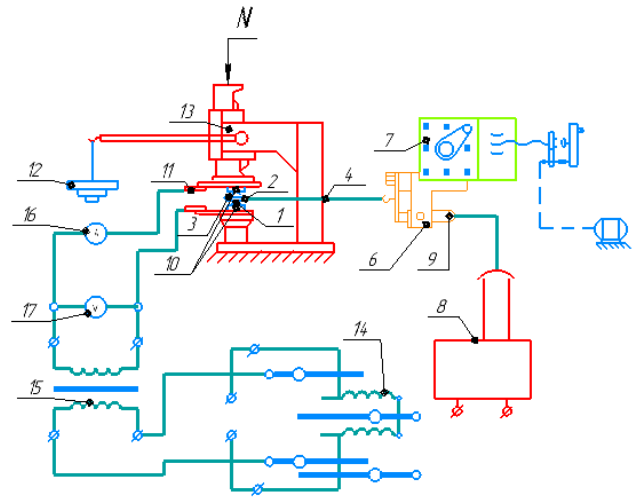


Fig. 2. (Color online) Adhesion testing facility.

In this case, such tribotechnical characteristics as the shear strength of adhesive bonds ( $\tau_{mn}$ ), normal contact stresses ( $p_{rn}$ ), the adhesion component of the friction coefficient ( $f_a$ ) were determined.

Experimental milling tests were carried out on the certified equipment to ensure the validity of the test results obtained. Tests were carried out on a vertical milling machine of normal accuracy and rigidity “VM 127M”. To investigate wear patterns of cutting tools at face milling with end cylindrical milling cutters (diameter  $d=12$  mm; number of teeth  $z=4$ ; with the geometry of the cutting part helix angle  $\omega=30^\circ$ ; front angle  $\gamma=10^\circ$ ; rear angle  $\alpha=4^\circ$ ) of grade H10F with various coatings, chromium-nickel alloys — EP99; EK61 were used as a machining material. Chemical composition, physical and mechanical properties and heat treatment of researched machined alloys are presented in GOST 5632-72.

The machinability of high-temperature chromium-nickel alloys in field tests was investigated according to the recommendations and regimes used by the companies in  $n=800$  rpm;  $S=65$  mm/min;  $a_e=4$  mm;  $a_p=1$  mm. In the process of milling to ensure the identity of results and to exclude errors of measurement the width  $h_r$  of tool wear chamfer on the back surface was measured by reference microscope MIR-2M with nozzle MOV-15 precision readout to 0.002 mm in the workplace and after reaching  $h_r=0.35-0.4$  mm for additional control and photofixation of the wear pattern a universal motorized stereo microscope with telecommunication capability “Carl Zeiss Stereo Discovery V12” with visualization system based on the video camera “Zeiss Axiocam 503 Color” 3 mp was used. The wear of the cutter was measured at specific numbers of passes, respectively the critical cutting path length to get a picture of all the stages of the wear curve (running-in, normal and catastrophic wear section).

Nanostructured composite multilayer coatings for adhesion tests on the tool material indenter and wear-resistant coatings for end mills were applied on three different units: “Platit  $\pi 311$ ” at the “Coating Technology and Heat Treatment” laboratory — (CrAlSi)N; (CrAlSi)N + DLC (Diamond-Like Carbon); (TiAlSi)N; (CrAlSi)N + epilama; (CrAlSi)N + DLC

+ epilama; (TiAlSi)N+ epilama; modernized HHB-6.6-II in laboratory “Innovative materials and coatings” IDTI RAS — (TiCrAl)N; (ZrCrAl)N; (ZrMoAl)N; (ZrMoHfCrAl)N and “Platit π411” at the Technopark “Vityaz” — TiB<sub>2</sub>; nACo<sub>3</sub>; nACRo; nACo<sub>3</sub>+TiB<sub>2</sub>; nACRo+TiB<sub>2</sub>; TiB<sub>2</sub>+ epilama; nACo<sub>3</sub>+ epilama; nACRo+ epilama; nACo<sub>3</sub>+TiB<sub>2</sub>+ epilama; nACRo+TiB<sub>2</sub>+ epilama at the coating thickness of no more than 10 μm was ensured (nACo<sub>3</sub> — (TiAlSi)N+TiN+TiAlSiN+TiN, nACRo — TiAlN is embedded in the amorphous matrix SiN). The grain size in these coatings was in the range of 3 to 5 nanometers. Microhardness for the nACo<sub>3</sub> coating was 45 GPa and for the nACRo coating 42 GPa, room temperature friction coefficient was 0.35 and 0.45, respectively.

#### 4. Results of experimental studies and their analysis

The analysis of works of foreign and domestic researchers allows us to conclude that the solution of the problem of increasing the wear resistance of cutting tools with different coatings is developing in different directions. At the same time there is practically no data on the results of experimental research with the indication of cutting modes, a particular pair “tool — machined material”, application modes and percentage content of elements of wear-resistant coating, as well as their operational characteristics (adhesion interaction coefficient, durability period, critical cutting path length and wear on the rear surface of the cutting tool), which does not allow one to conduct a comparative analysis of the obtained results. In the works [20, 21] to solve the problem we went along the way of modeling alloying components (Ti, Cr, Al, Si) in the composition of nanocomposite coatings (nc-TiAlN/a-SiN, nc-AlCrN/a-SiN, nc-ALTiCrN/a-SiN), thus an increase in the tool wear resistance on the average by 10–15% does not allow one to ground improvement of tribotechnical characteristics of wear resistant coatings, considering possible errors of measurements.

At the same time, it is known [23–27] that most modern nanostructured multilayer composite coatings used on the cutting tool for blade cutting machining under certain contact processes and temperature-force conditions can improve their tribotechnical characteristics. In particular:

- The nanocomposite coating based on aluminum nitride and titanium nACo<sub>3</sub> is a high-tech hard coating applied by

physical vacuum deposition and it consists of nanoparticles residing in a binding amorphous matrix. The uniqueness of the coating lies in the successful combination of physically mutually exclusive parameters: while increasing the hardness its elasticity simultaneously increases;

- The nanocomposite coating based on chromium and aluminum carbonitrides nACRo with very high hardness at elevated cutting temperatures;

- Titanium diboride TiB<sub>2</sub> nanocomposite coating — a synthetic particularly hard, heat-resistant, refractory and wear-resistant material. These coatings are advantageous due to their high hardness and modulus of elasticity as well as good abrasion resistance;

- DLC (Diamond-Like Coating) is a diamond-like carbon material with a strong and friction-reducing coating applied over other conventional wear-resistant coatings in most cases. Graphite C + 10–20% impurities.

The results of adhesion studies are shown in Figs. 3, 4. The analysis of experimental data showed that with increasing contact temperature, the adhesion component of the friction coefficient increases monotonically for all the coatings under study, at temperatures corresponding to the optimal cutting speeds (according to the wear intensity), the lowest value corresponds to the most favorable composition and technology of their application. It was found that:

- The most effective coatings in terms of contact processes on a single contact spot and the adhesion coefficient are the coatings applied on the installation “Platit π411”: nACo<sub>3</sub>; nACRo; nACo<sub>3</sub>+TiB<sub>2</sub>; nACRo+TiB<sub>2</sub> and (CrAlSi)N+DLC;

- The lowest coefficient of adhesion interaction between the tool material and the machined material over the entire temperature range under study is provided by such coatings nACo<sub>3</sub>+TiB<sub>2</sub> at EP99 and nACRo+TiB<sub>2</sub> at EK61, which confirms the best adhesion of the coating with the substrate and allows their use for high-speed milling.

The results of experimental studies of the wear resistance of cutting tools with different coatings during milling (Fig. 5–8) of chromium-nickel alloys are presented as graphs of the dependence of tool wear on the back surface ( $h_p$ , mm) of the critical cutting path length ( $l$ , m) (Figs. 5, 7), as well as the diagram of dependence of the durability period on the coating applied to the cutting tool (Figs. 6, 8). The best indices of cutting tool wear resistance (wear on the back surface, critical cutting path length and tool durability period) were provided:

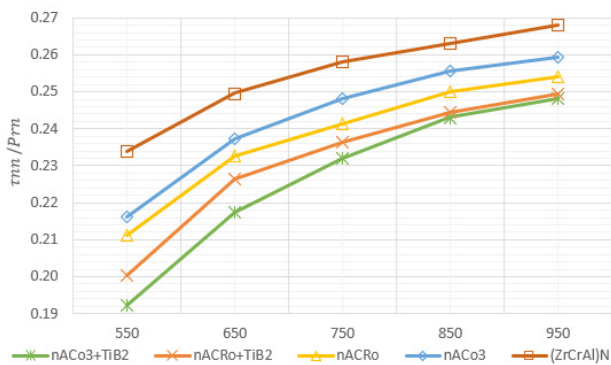


Fig. 3. (Color online) Temperature dependence of frictional characteristics of plastic contact “EP99-H10F with coatings”.

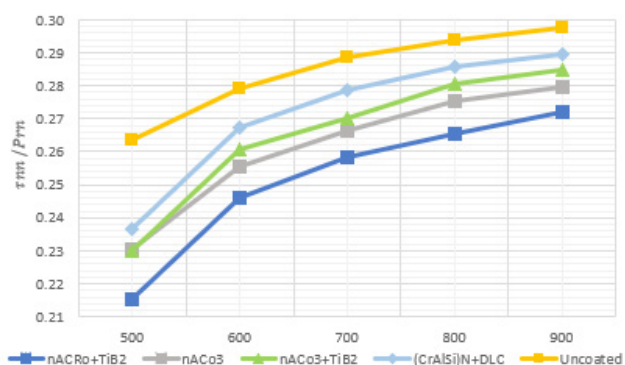
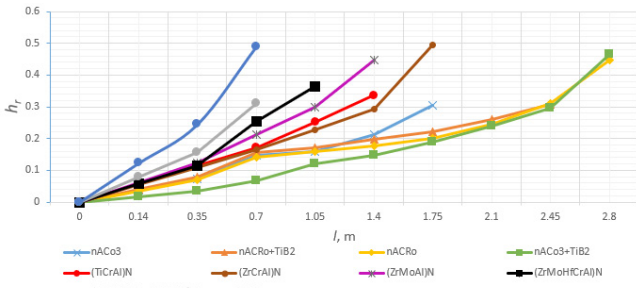
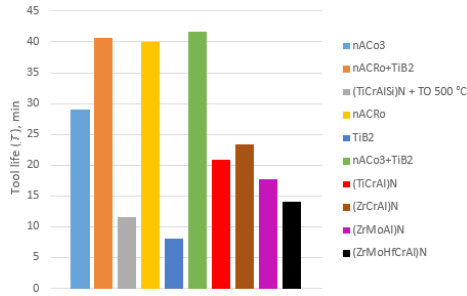


Fig. 4. (Color online) Temperature dependence of frictional characteristics of plastic contact “EK61-H10F with coatings”.



**Fig. 5.** (Color online) Influence of rear surface wear rate during milling of EP99 chromium-nickel alloy with H10F carbide cutters with different coatings at  $n = 800$  rpm,  $S_{min} = 65$  mm/min,  $a_c = 4$  mm,  $a_p = 1$  mm.



**Fig. 6.** (Color online) Tool life when milling chrome-nickel alloy EP99 with H10F carbide cutters with different coatings at  $n = 800$  rpm,  $S_{min} = 65$  mm/min,  $a_c = 4$  mm,  $a_p = 1$  mm.

- at milling of chromium-nickel alloy EP99 when using wear-resistant coatings: improvement by 24% with “ $nACo_3 + TiB_2$ ” coating and by 23% with “ $nACRo + TiB_2$ ” coating as compared to the coating “ $nACo_3$ ”;

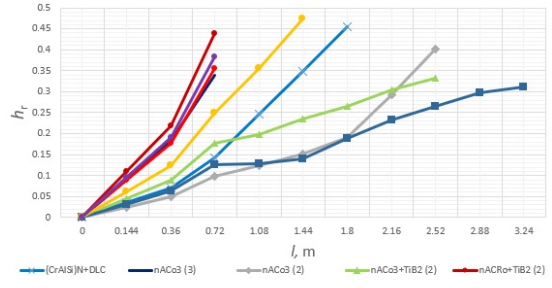
- at milling of chromium-nickel alloy EK61 when using wear-resistant coating: improvement by 23% with coating “ $nACRo + TiB_2$ ” in comparison with coating “ $nACo_3$ ”.

To explain the mechanism of increasing tribotechnical characteristics of multilayer nanostructured coatings (Fig. 9), a series of material science studies of well samples of machined and spherical surface of tool material were conducted.

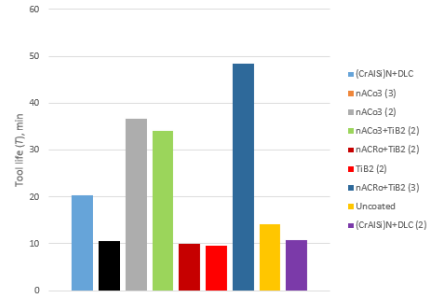
The surface morphology and microstructure of the coatings were studied by transmission electron microscopy on a JEOL JEM-201 OE with an accelerating voltage of 200 kV. At the same time, the coatings were deposited on the cemented carbide bottom layer with a thickness of 2.5 to 3  $\mu$ m for observations. Samples were prepared using the FIB ion beam focusing method on the JEOL-JFIM-2100 system and were thinned to 0.1  $\mu$ m using Ga ions with an accelerating voltage from a Ga ion source of 30 kV and a current of 2.0 mA. The



**Fig. 9.** (Color online) Samples from machinable (heels) and tool (indenter with different coatings) materials.



**Fig. 7.** (Color online) Influence of rear surface wear rate during milling of EK61 chromium-nickel alloy with H10F carbide cutters with different coatings at  $n = 800$  rpm,  $S_{min} = 65$  mm/min,  $a_c = 4$  mm,  $a_p = 1$  mm.

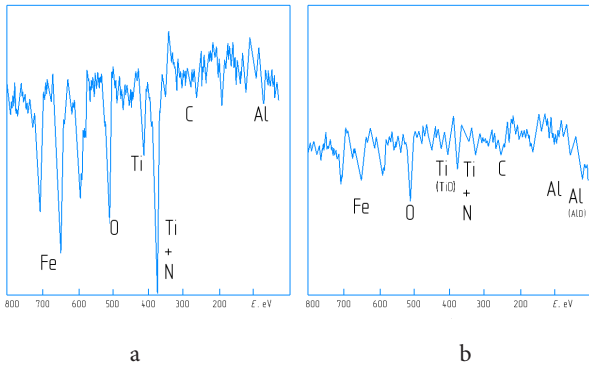


**Fig. 8.** (Color online) Tool life when milling chrome-nickel alloy EK61 with H10F carbide cutters with different coatings at  $n = 800$  rpm,  $S_{min} = 65$  mm/min,  $a_c = 4$  mm,  $a_p = 1$  mm.

chemical composition of the secondary structures arising on the cutter surface during cutting was investigated by the VIMS method. The ion etching rate was on the order of 0.2 monolayer per minute and the analysis was performed in static mode. The atomic structure of the films formed on the tool surface during cutting was investigated using EELFS and an ESCALAB MK 2 (VG) electron spectrometer. The friction surface was investigated in areas free from adhesion of the workpiece material. A high magnification ratio (2000 $\times$ ) was used. The primary electron energy was  $E_r = 1000$  eV. A fine spectral energy loss pattern was recorded close to the elastically scattering electron line in the 250-eV range. The conditions for the analysis were chosen to provide the best energy resolution with a good signal intensity ratio.

Intensive tribo-oxidation of the cutting tool surface occurs during high-speed machining. Fig. 10 shows Auger-electron spectra for tools with  $nACRo$  wear-resistant coating. The oxidation of the contact surfaces is obvious, as evidenced by the presence of large amounts of oxygen in both spectra. The intense ionic peaks correspond to the adhesion zones of the part material. The Ti line is significantly stretched (Fig. 10b) in this zone; this is the result of the oxidation process. An increased amount of aluminum oxide is observed in the spectrum of the filtered coatings, which is shown as a shift in the aluminum line to a lower energy zone (60 eV). At the same time, the intensity of the metallic Al LMM line level around 68 eV decreases.

Figure 11a–b shows a series of spectra of positive secondary ions for both 3 minutes of cutting and after 20 minutes of cutting with the coating and Figure 11c–d shows spectra of negative secondary ions for both coatings. In both positive secondary spectra, the TiO line intensity is high and this is due to intense tribo-oxidation, which forms

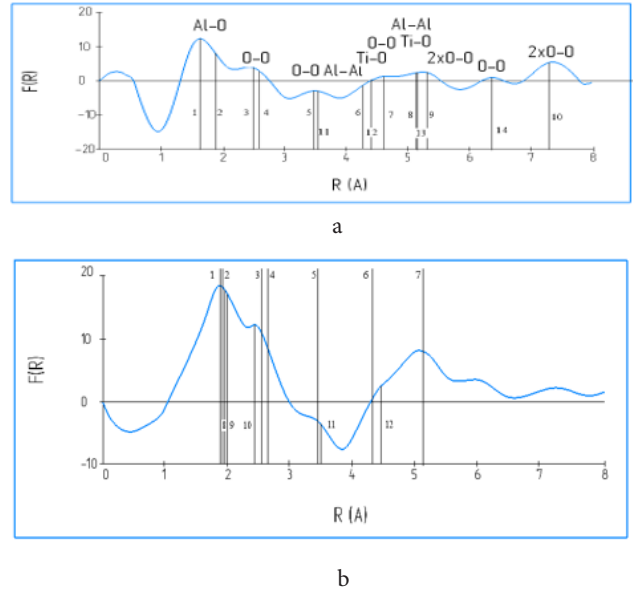


**Fig. 10.** Auger-electron spectra of surfaces of worn *nACRo*-coated inserts: at the running-in stage after 3 minutes from the beginning of cutting (a); at the steady-state stage after 20 minutes from the beginning of cutting (b).

rutile-like films. But some aluminum oxide is formed only on the surface of the coatings after 20 minutes and this effect can be observed on the spectrum of negative secondary ions (Fig. 11c). The formation of aluminum oxide films on the surface of the cutter significantly changes the heat fluxes and heat removal to the chip. This is confirmed by images of chip cross sections after scanning on an electron microscope, and three different zones can be seen in the chip cross sections.

The atomic structure of the films appearing on the wear zone surface during tribooxidation of the *nACRo* coating was compared with the oxide layer obtained during oxidation of the binary TiAl alloy under equilibrium conditions. Figure 12 shows the Fourier transforms as a result of mathematical consideration of the fine structure of electron spectra close to the line of elastically scattering electrons. The positions of the peaks on the Fourier transforms correspond to the interatomic distances for the closest coordination spheres. The decoding of the data was based on the analysis of known crystal characteristics of oxides formed on the surface. The positions of the main peaks of the Fourier transforms correspond to the interatomic distances associated with the  $Al_2O_3$ , and  $TiO_2$  lattices (Fig. 12).

In general, the results of metallurgical studies allow us to state that the use of such coatings contributes to the reduction of friction forces and cutting tool wear due to the formation of titanium and aluminum oxides. Studies of the protective film formation at low and moderate cutting speeds show that there is only one type of the protective film formed on the surface



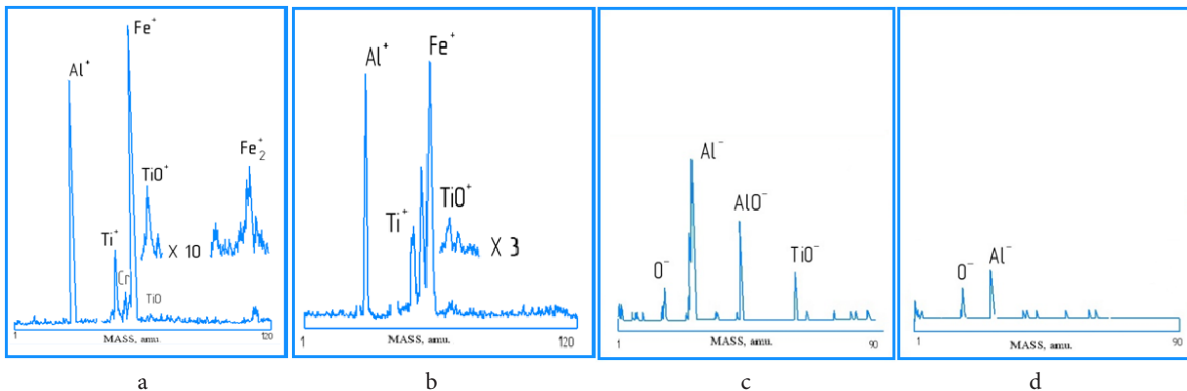
**Fig. 12.** Fourier transforms on the EELFS close to the elastic scattering electrons for films formed on the surface: *nACRo* coated cutting tool (a); binary TiAl alloy after isothermal oxidation in air (b).

as a result of self-organization phenomena [9,10,26]. These films have an amorphous-like structure [9,10,26] with high ductility and improved lubricity. More complex phenomena occur during high-speed processing. These are primarily the low-intensity peaks found at the far atomic distances on the Fourier transforms shown in Fig. 12. From this figure, it can be assumed that the films that form during tribo-oxidation under high-speed processing conditions are amorphous-like. These films of aluminum oxide contribute to the reduction of wear, because due to the low thermal conductivity they prevent the intensive removal of heat generated during cutting into the body of the cutting tool.

### 5. Conclusions

According to the results of experimental studies, it was found that:

1. The best adhesion performance of EP99-H10F is achieved with:  $nACo_3 + TiB_2$  and  $nACRo + TiB_2$  coatings, and for the pair EK61-H10F — with “ $nACRo + TiB_2$ ” and “ $nACo_3$ ”;
2. When milling chromium-nickel alloys with cutting tools with nanostructured multifunctional coatings, the



**Fig. 11.** Spectra of positive (a, b) and negative (c, d) secondary ions of the surface of worn *nACRo*-coated plates: after 3 minutes of cutting (a, d); after 20 minutes of cutting (b, c).

improvement of wear resistance (wear on the rear surface, critical cutting path length and tool life period) was provided:

- when using “ $nACo_3 + TiB_2$ ” coating by 24% and using “ $nACRo + TiB_2$ ” coating compared to “ $nACo^3$ ” coating by 23% when machining EP99 alloy;

- when using “ $nACRo + TiB_2$ ” coating by 23% compared to “ $nACo^3$ ” coating when machining EK61 alloy.

3. The above effects, according to the studies both by domestic [9,10] and foreign researchers [26,27], as well as by ours, can be explained by the adaptation of friction surfaces to external temperature and force effects, in which protective amorphous and lubricating films are formed.

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