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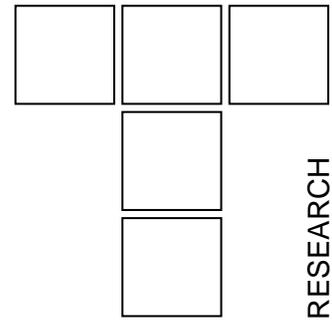V. POPOV, A. LUBCHENKO, A. OLEJNIK

RESEARCH

# The Tribological Investigations of Multicomponent Multilayered Ion-Plasma Coatings Avinit

*The friction and wear characteristics of sliding friction in conditions of boundary lubrication for coatings, received by ion-plasma method on the heat resistant deformed alloy of AK4-1 type are investigated. Conducted tribological studies of improved designs of multi-component multi-layer coatings Avinit in fuel TS-1 media selecting coating materials for precision friction pairs of hydraulical units. Coatings are very effective in increasing the stability of the pair to bores cuffing teasers. Improved coatings have low friction coefficients (0.075 - 0.095) at loads up to 2.0 kN and showed high resistance to wear. More over relative stability increasing achieve 20-80 times, and contrabody wear decrease of 4-5 times.*

***Keywords:*** *vacuum-plasma coatings, nanolayers and multilayers, coatings, tribology.*

## 1. INTRODUCTION

Modern researches in the field of creation of new materials with record characteristics of wear resistance, roughness, a possibility to work in extreme conditions are connected with an area of nanotechnologies, which allow forming multi-component compositions with structural elements having sizes from several hundreds to units of nanometers. Such materials in comparison with materials of the same composition with a regular structure can have several times higher corresponding characteristics of tribological and other properties. It also concerns coatings – one of efficient methods of extension of possibilities of application of one or another material [1 - 7].

In works [8, 9] we have conducted researches on coating multi-component multilayer and nanolayer strengthening coatings on the basis of titanium, molybdenum and their compounds with nitrogen using methods of vacuum-plasma deposition. Multi-component multilayer coatings showed higher indexes of wear resistance and tribological characteristics compared with one-layer coatings on the basis of one compound.

In works [10, 11] there is described created by us experimentally-technological equipment - *Avinit* device for coating multilayer functional coatings.

Its essential distinctive feature is that the device allows to implement complex methods of coating functional coatings (plasmachemical CVD, vacuum-plasma PVD (vacuum-arc, magnetron), processes of ionic saturation, implantation and ion-beam processing of a surface), united within one technological cycle.

Substantial growth of a spectrum of the sources, caused by composite nature of used methods, allows to obtain coatings practically from any elements and alloys, high-melting oxides, carbides, nitrides, ceramic-metal compositions on the basis of high-melting metals and oxides, that essentially expands possibilities of creation of absolutely new materials and coatings of nodes and details with different functions, working in extreme conditions considering temperature, influence of corrosive mediums, mechanical loadings.

The second distinctive feature of *Avinit* device is a possibility to pass on to nano-scale for implementation of processes of controllable formation of multi-component nano- and microstructural coatings with the given characteristics.

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It became possible thanks to the conducted radical reorganization of control over operation of all systems of technological equipment on the basis of the technology of open synchronization of operation of the systems of ion-stimulated deposition and diagnostics equipment of nano-scale coatings at the expense of introduction of new microprocessor supply systems to the equipment, synchronization and control over synthesis and diagnostics processes and development of a complex of controlling methods over technological parameters in the course of coating for purposeful control over the technological process.

There is a possibility to form multilayer structures containing a great number of layers of a various chemical compound (metal, nitride, carbide, oxide, etc.) being thick from units to hundreds of nanometers. These layers are put in *Avinit* device with usage of combined methods – PVD (vacuum-arc and magnetron sputtering) and CVD (gas-phase and plasmachemical sedimentation). The structure of layers is secured by programmed coordinated operation modes of sources of plasma (both PVD and CVD), working gases and high potential applied to a substrate.

A correct choice of individual materials of layers, methods of sedimentation and optimization of technological parameters create premises of synthesis of materials with a complex of unique properties, including exceptionally high hardness, durability, chemical stability, low coefficient of friction and raised wear resistance.

Within operational frameworks [11] a number of hardware and technological developments (application of advanced separating devices, improved diagnostics of plasma and gas flows, perfection of IR measurements (in an infrared range) of temperature fields of the covered products, perfection of mechanical and electronic systems of protection against microarcs and modernization of cathode nodes and management system) was implemented, that allowed to expand essentially possibilities of technology equipment and to provide coating of qualitative coatings on precision surfaces.

Coatings of *"Avinit"* type fall out on precision surfaces with high-class purity up to 12-13 class without lowering the class of purity of a surface. This is reached by a possibility to use efficient methods of cleaning a surface in developed technologies – cleaning in glow-discharge Ar, cleaning in two-stage vacuum-arc discharge and cleaning by metal ions under pressure above zero point of growth, and also prevention of damage of a surface by microarcs, for that in "*Avinit*" device there is a three-level system arc suppression, providing high quality of cleaning of a surface from oxides and other pollution without appearance of electrical breakdowns. Deposition happens at low temperatures, not exceeding temperatures of tempering of material of a basis, providing preservation of mechanical characteristics and absence of warpage of coated products.

The conducted reconstruction of technology equipment and the developed software solutions allowed to move to a qualitatively new level on further modification and perfection of constructions of functional coatings of *Avinit* type, stability of technologies and increase in control over their quality while coating such coatings for developed pairs of friction for possible use in details of precision friction pairs.

In this work the results of metallographic and tribological researches of improved coatings of *Avinit* type on the basis of Ti-Al-N system and on the basis of Mo-N system are presented.

## 2. METHODS OF RESEARCHES

### 2.1 Method of Obtaining Coatings "*AVINIT*"

For obtaining multilayer coatings "*Avinit*" from solids in Ti-Al-N system a technological two-cathode circuit was used with simultaneous operation of two sources of sputtering which are allocated towards each other in the environment of reactionary gas with rotation of the sample around its axis, and for coating multilayer coatings based on the sequence of solid and soft layers (TiN-Ti, MoN–Mo systems), - a one-cathode circuit with continuous operation of a source of sputtering and impulse (periodic) submission of reactionary gas.
Coatings were put on samples of widely used in engine production and aggregate production materials from steel DIN 1.2379 (x12Ф1) with hardness 56 … 61HRC with precision surfaces ($R_a$ = 0,016-0,021 microns (12-13 class of roughness) and on samples from aluminum alloy DIN 7980 (AK4-1).

### 2.2 Method of Metallographic Researches

Metallographic researches and determination of parameters of materials (thickness of coatings, uniformity, deficiency and structure of the material)

were done on microscope MMR-4. Microhardness of coatings was defined by means of microhardness tester PMT-3 under loading 50 H. Hardness of material was measured on the hardness tester by means of pressing a diamond tip by Rockwell method. Roughness of the surface of samples before and after coating was measured by profilometer-profilograph.

Measurements of microhardness and Young's modulus in multilayer and nano-layer coatings of *Avinit* type 1 … 3 microns were carried out with an instrument for measurement of nano-hardness by CSM company (Switzerland) (speed of loading 20,00 mH/min, max depth 100,00 nm under loading 0,6 H processing of results in Oliver–Far model).

Carrying out of researches of chemical individuality of a subsurface area of functional coatings was fulfilled with a method of mass-spectrometry of secondary ions (SIMS), electron X-ray microanalysis (EXMA), scanning electronic microscopy (SEM). Removal of profiles of change of chemical individuality of nano-layers of functional coatings was carried out by means of a method of mass-spectrometry of secondary ions (SIMS) on secondary-issue mass analyzer MA 7201M. The maximum depth of profiling is 5 microns. For sputtering the bundle of Ar $^{+}$ ions with energy 5–7 keV was used. Research of functional sections of a surface of samples was carried out by means of scanning electronic microscopy (SEM). Removal of space allocations of chemical elements was carried out by means of electron X-ray microanalysis (EXMA). Metallophysic measurements of the obtained coatings were carried out on scanning electronic microscope JSM T-300.

### 2.3 Method of Research of Characteristics of Friction And Wear

Tribological tests of antifriction, wear properties and seizure of samples with coatings were carried out by the machine of friction 2070 SMT-1 under the circuit "cube" - "roller" with a graduated loading in the range of loadings 1-20 MPa. Tests were done in the environment of TS-1fuel and in the environment of diesel fuel. For determination of seizure of surface layers of materials of pairs of friction loading was done from $P_{min}$ to critical value of $P_{cr}$ under which seizure happens.

In the process of tribological tests there were registered values of force of friction $F_{fr}$, normal loading $N$, contact pressure $P$, values of which helped to judge about mechanical losses in tribosystems. Friction coefficients were defined as $f = F_{fr} / N$.

## 3. EXPERIMENTAL RESULTS

One of indexes of multilayer coatings, in many respects defining their properties, is thickness of a separate layer. While a coating is being formed the necessary thickness of a layer is set by an operating time of a corresponding source, which demands knowledge of growth rate. Growth rate of a coating generally depends on capacity of a source of sputtering, distance from a source to a substrate, orientation and its position in relation to an axis of the direction diagram of an atomic flow of a source of sputtering, the form of the direction diagram, potential of displacement affecting the substrate. The substrate can be fixed, rotate around the fixed axis or make planetary motion.

In tab. 1 results of experiments on determination of growth rate of various coatings received both on fixed substrates, and on the substrates making planetary motion, are shown.

**Table 1.** Growth rate of coatings of various composition.

| № art. | Coating | Growth rate, V, micron/hour | Remarks |
|---|---|---|---|
| 1 | Ti | 0,25 | Planetary motion |
| 2 | Mo | 0,2 | -----x------ |
| 3 | TiN | 0,16 | -----x------ |
| 4 | MoN | 0,14 | -----x------ |
| 5 | TiAlN | 0,7 | |
| 6 | TiN | 0,9 | Fixed position |
| 7 | MoN | 0,7 | -----x------ |

On the basis of the data about growth rate of coatings the data for several variants of nano-layer coatings were entered in the control program of *Avinit* device, namely:

- coatings Ti-TiN with the period of recurrence of 10 nanometers and thickness of separate nano-layers correspondingly 2 nanometers and 8 nanometers;

- coatings Mo-MoN with the period of recurrence of 20 nanometers and equal thickness of separate nano-layers;

- coatings TiN-AlN with the period of recurrence of 12 nanometers and thickness of separate nano-layers 4 and 8 nanometers.
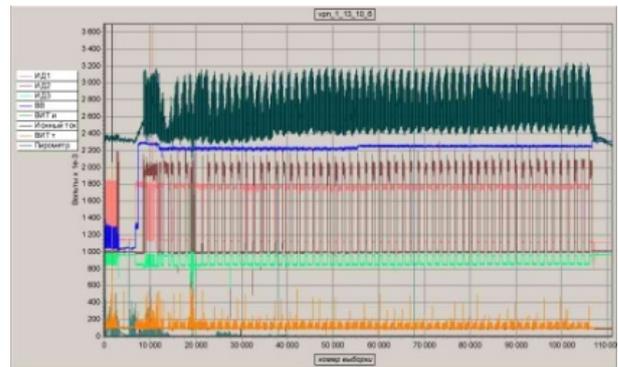
Protocols of the automated monitoring system of these processes are presented in Fig. 1.
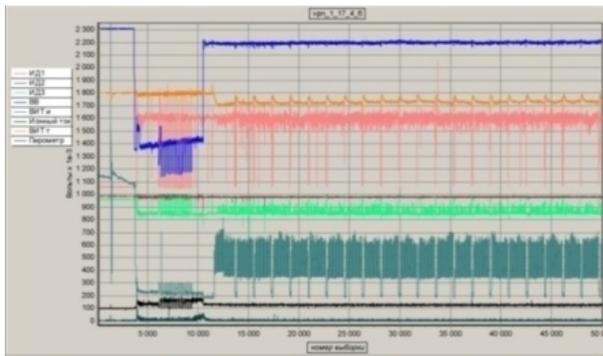


a)
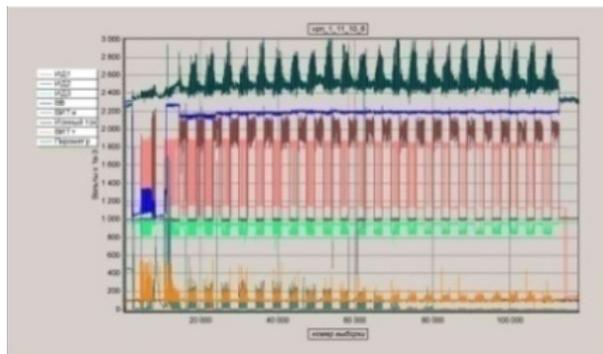


b)



c)

**Figure 1.** Protocols of the automated monitoring system of the process of obtaining of coatings:
a) Ti-TiN with the period of recurrence of 10 nanometers and thickness of separate nano-layers 2 nanometers and 8 nanometers;
b) Mo-MoN with the period of recurrence of 20 nanometers and equal thickness of separate nano-layers;
c) TiN-AlN with the period of recurrence of 12 nanometers and thickness of separate nano-layers 4 and 8 nanometers.

The composition and some characteristics of hardness, microhardness and roughness of the tested coatings received by means of various technological circuits, are shown in tabl. 2.

**Table 2.** Characteristics of samples

| N art | Composition of coating | Initial parameters | | Finite parameters | | |
|---|---|---|---|---|---|---|
| | | Hardness of a basis, HRC | Microhardness of the surface of a sample, $N_v$ (MPa) | Thickness of coating, micron | Microhardness of the surface of a sample with coating, $N_v$ (MPa) | Roughness, $R_a$, micron |
| 1 | Without coating | 59 – 60 | 770 - 800 | | | 0,025 (12b) |
| 2 | TiN | 59 – 60 | 770 - 800 | 1,0 | 1500-1900 | 0,040 (11c) |
| 3 | TiN-Ti | 59 – 60 | 770 - 800 | 1,0 | 1300-1800 | 0,036 (12a) |
| 4 | MoN | 59 – 60 | 770 - 800 | 1,0 | 2000-2300 | 0,040 (11c) |
| 5 | MoN-Mo | 59 – 60 | 770 - 800 | 1,0 | 2000-2200 | 0,036 (12a) |
| 6 | TiN-AlN | 59 – 60 | 770 - 800 | 1,0 | 2600-3500 | 0,025 (12b) |
| 7 | MoN (without a separator) | 59 – 60 | 770 - 800 | 10,0 | 2000-2200 | 0,60 (8a) |
| 8 | TiN (without a separator) | 59 – 60 | 770 - 800 | 12,0 | 1500-1800 | 0,70 (7c) |

Comparing roughness of a substrate and coating shows that after coating samples with roughness equal to 12-13 classes of purity, roughness of the surface practically does not change, or there is an insignificant increase in the roughness which practically does not fall outside the limits of one class according to the classes of roughness of a surface. The conducted X-ray researches of coatings of *Avinit C/P 320-n1* type showed that coatings in their composition have ~ 45 at % Al.

The crystalline structure corresponded to TiN structure with grid parameter close to values of this connection. According to X-ray researches the size of areas of coherent scattering (ACS) in the coating made 32 nm. This value is well coordinated with the sizes separate nano-layers TiN and AlN (nano-layer growth rate for one turn ~ 35 nm), that confirms presence of a nano-layer structure according to the technological circuit of formation of coating.

In fig. 2 the results of electron X-ray microanalysis (EXMA) of *Avinit* coating *C/P 320-ms1* for three elements are shown: aluminum, iron and titan on the sample: *Avinit C/P 320-ms1* at scanning by an electronic bundle of the sample (diameter of an electronic probe Ø=30 nm, characteristic radiation is registered in the surface layer of a sample at the depth of 1 micron).
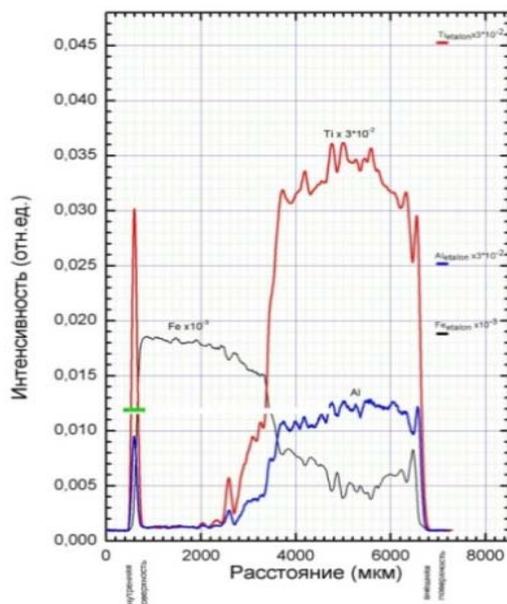


*a)*
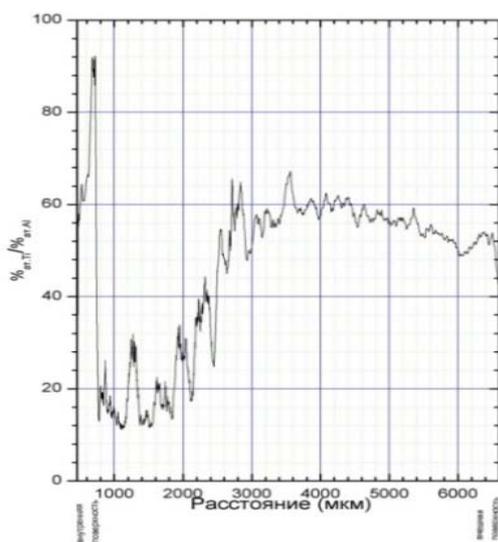


*b)*

**Figure 2.** Allocation of characteristic X-ray radiation of atoms of elements in coating *Avinit C/P 320-ms1 – à). % at. Ti / % at. Al – b).*

While scanning from the exterior surface to the interior one curves begin with peaks of intensity of titan and aluminum caused by characteristic radiation of these metals, placed on the exterior cylindrical part of a sample. Quantities of peaks of characteristic radiation

Al and Ti with intensities of the same order are also observed on the conjunction of interior face and cylindrical surfaces. Allocations Al and Ti all over the analyzed surface are qualitatively close to each other.

In the surface with unaffected functional coating, despite existence of coating, there is observed characteristic Fe radiation, which intensity is much less, than that from Al and Ti. The type of dependence of a signal on Fe has an opposite character in comparison with them. In fig. 2b), where allocation of relation of quantity of atoms Ti to quantity of atoms Al is shown, it can be seen along the analysis line that the quantity of titan is a little bigger than that of aluminum.



x9500       a
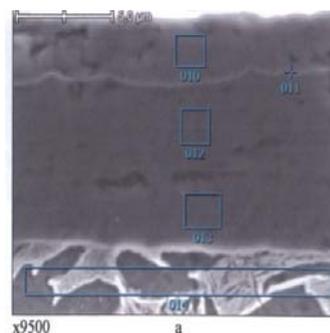
| № точки | N | Al | Ti | Mo | Всего,% |
|---------|------|-------|-------|------|---------|
| 010 | 9,10 | 27,93 | 62,96 | | 100 |
| 011 | 6,89 | 16,73 | 76,38 | | 100 |
| 012 | 10,7 | 45,87 | 43,44 | | 100 |
| 013 | 10,71 | 47,22 | 42,07 | | 100 |
| 014 | | 3,64 | 88,94 | 7,41 | 100 |

б

**Figure 3.** External view of coating *Avinit C/P 320-n1* (cross-sectional view) with marked zones of analysis–a); approximate chemical compound of analyzed zones–b).
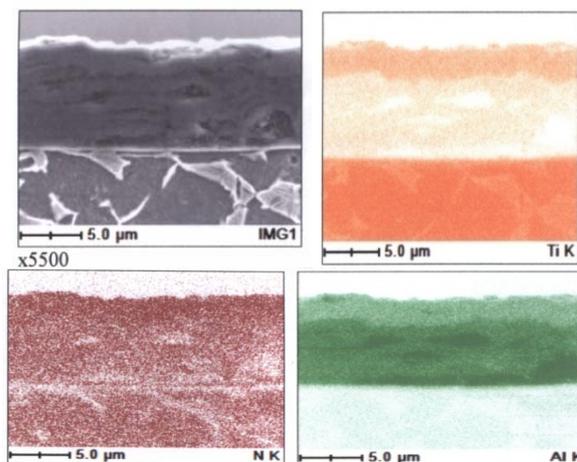
Thickness of coating ~ 9 microns.



**Figure 4.** External view of coating *Avinit C/P 320-n1* (cross-sectional view) in a mode of mapping a part of coating. Bigger concentrations of elements correspond to more intensive coloring.

Results of metallophysic measurings of coating *Avinit C/P 320-n1* on scattering electronic microscope JSM T-300 are shown in fig. 3, 4.

Thickness of thick non-filtered multilayer coatings *Avinit C/P 100-t10* and *Avinit C/P 220-t10* - 10 … 15 microns, Measured values of hardness of coatings of *Avinit C/P 320-ms1* type constituted not less than $N_V$ = 3500 kNH/mm$^2$, coatings of *Avinit C/P 210-m1* type - not less than $N_V$ = 2000-2500 kH/mm$^2$.

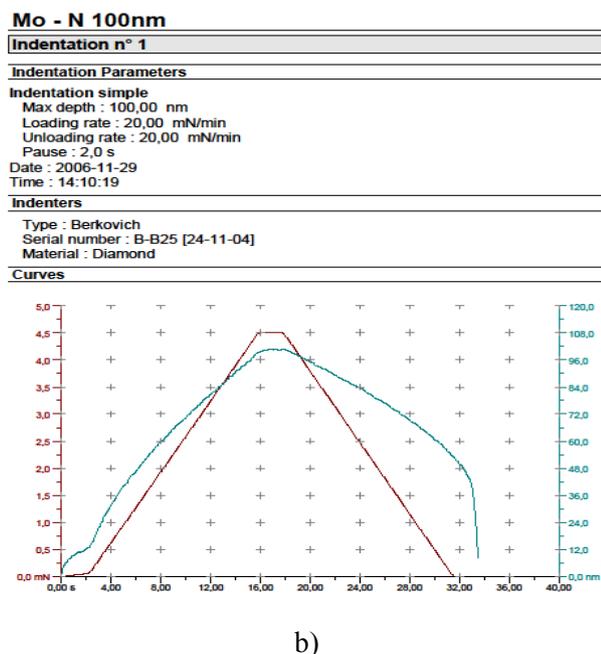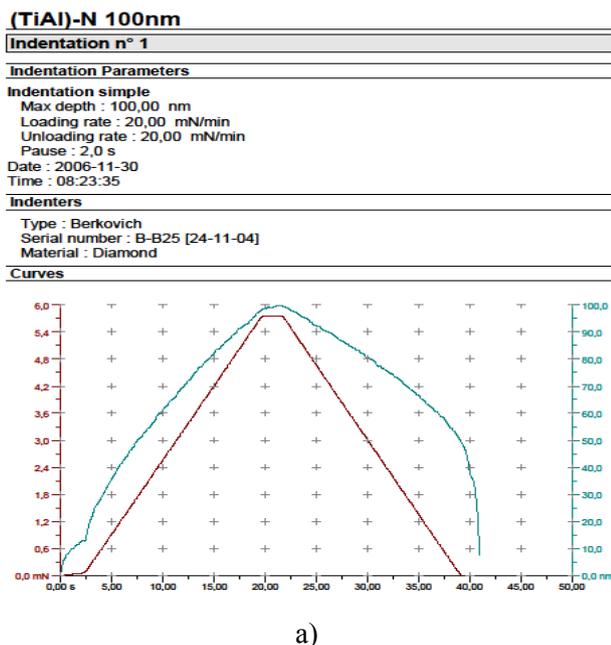Thickness of thin multilayer and nano-layer coatings of *Avinit C/P 320-ms1* type and *Avinit C/P 210-m1* - 1..2 microns. For determination of microhardness of thin coatings (<4 microns) nano-hardness was measured with the help of nano indentation tester by CSM company (Switzerland) (loading rate 20,00 mH/min, max depth 100,00 nm at the level of loading - 0,6H), processing of results with usage of standard software on the basis of application of Oliver–Far model. The conducted measuring of microhardness and Young's modulus in coatings *Avinit C/P 320-ms1* with thickness of 1,4 microns resulted in the following values: $N_v$ = 1600 – 2300 kH/mm$^2$, $E$ = 250 - 300 GPa, coefficient of Poisson's ratio $K$ = 0,30 (diagrams of loading are shown in fig. 5a).

Similar measurements on coatings *Avinit C/P 210-m1* with thickness of 1,0 microns showed the following results - $N_v$ = 1500 – 1800 kH/mm$^2$, $E$ = 200 - 260 GPa, Poisson's ratio $K$ = 0,30 (fig. 5b). It is necessary to mark that in model of Oliver-Far Young's modulus of coating and basis are assumed to be the same and consequently the calculated values can be a little lowered.
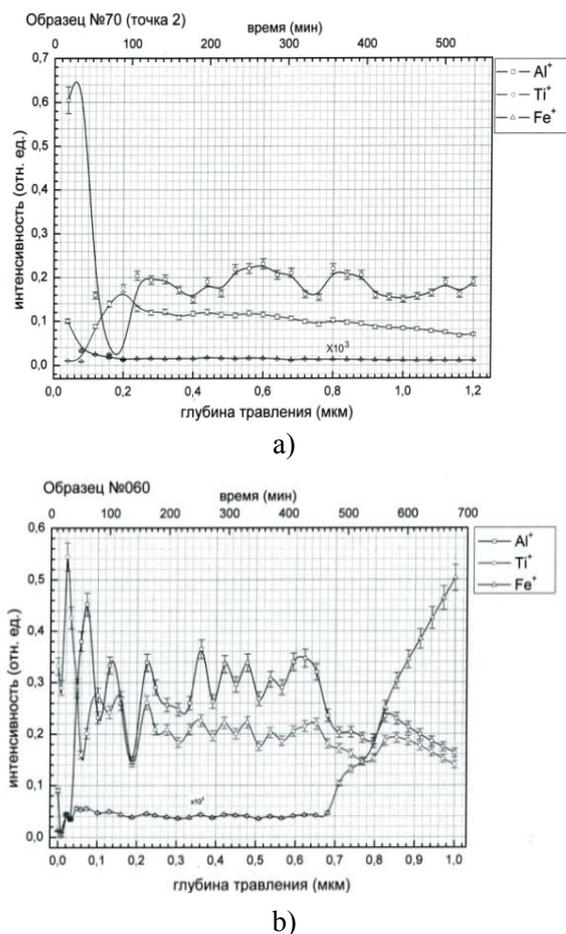


a)



b)

**Figure 5.** Measurements of nano-hardness and Young's modulus:
a) coating ***Avinit C/P 320-ms1***,
b) coating ***Avinit C/P 210-m1.***



a)



b)

**Figure 6.** Dependences of currents of secondary ions Al$^+$, Ti$^+$ on sputtering time:
a) coating Avinit C/P 310-ms1,
b) - coating Avinit C/P 320-n1.

Metallographic researches of coatings of *Avinit* type with application of methods of mass-spectrometry of secondary ions (SIMS), electron X-ray microanalysis (EXMA), scanning electronic microscopy (SEM) were conducted.

In fig. 6a) for coating *Avinit C/P 310-ms1* dependences of currents of secondary ions $Al^+$, $Ti^+$ on sputtering time and correspondingly depths of a profile of allocation of components are shown.
Change of the current of secondary ions for both experiments characterizes the change of concentration of corresponding elements deep into the sample in the process of sputtering of a subsurface area with a bundle of primary ions $Ar^+$. From the received dependences it follows that the exterior layer of coating has raised concentration of aluminum which with depth decreases. Similar dependences on profiles of allocation of aluminum and titan in a subsurface zone of the sample with functional coating *Avinit C/P 320-n1* are shown in fig. 6b). Synchronous changes of intensities of $Al^+$ and $Ti^+$ currents at depth ~ 1,8 microns from the surface are connected with technology of formation of coating.

Thus, experimental results confirm the possibility of low-temperature coating of highly rigid coatings *Avinit C* on the basis of nitrides of metals in the modes providing good adhesion to the material of a substrate (steel H12MF with precision surface $R_a$ = 0,025 microns) without essential decrease in steel strength characteristics (<200ºC) and without deterioration of the class of purity of original surface.

## 4. RESULTS OF TRIBOLOGICAL TESTS

In fig. 1. the results of tribological tests (coating on coating) ("cube-roller" circuit) of multilayer coatings *Avinit C/P 320-ms1* (micro) and *Avinit C/P 510 µ1* with counterbodies with multilayer coatings *Avinit C/P 220-t10* and *Avinit C/P 100-t10 (*on the basis of *TiN-Ti)* with thickness of 10–15 microns, precipitated from non-filtered plasma flows.

In tabl. 3 values of weight wear of samples during 8 hours of wear tests of the tested pairs of friction with multilayer coatings *Avinit* are shown.

Presence of coatings essentially increases resistance of tribopairs against teasers, raising values of $P_{cr}$ of teasers formation and practically preventing teasers formation.
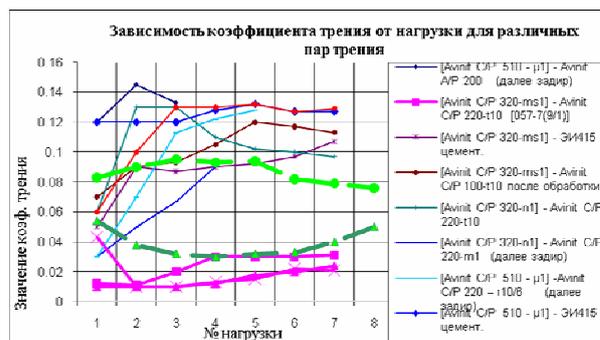


**Figure 7.** Dependence of coefficient of friction on loading.

**Table 3.** Values of wear of samples in the course of wear tests during 8 hours.

| Pair of friction | *Avinit C220-t (16 … 20)/10, ▼ 10 / Avinit C320-ms1* | | |
|---|---|---|---|
| **Wear**, gr Time of tests 480 min. | *Avinit t (16 … 20)/10* | *Avinit C320-ms1* | total |
| | fixed | mobile | 0,00091 |
| | 0,00091 | 0 | |
| | mobile | fixed | 0,00101 |
| | 0,00087 | 0,00014 | |

Especially efficient are coatings on the basis of *Avinit C/P 220-t10* which have the highest values of $P_{cr}$ and the lowest values of coefficient of friction. This is proven not only by maximum loading increase during tests, but also by a course of dependence of coefficients of friction on loading which after some loading increase up to 0.6-0.8 kN went down to the maximum loading 2 kN. Application of multilayer coatings (for example, *Avinit C/P 110* types *TiN-Ti)* leads to increase in $P_{cr}$ in comparison with monolayer coatings (for example, *Avinit C/P 100* type *TiN)*.

Coefficients of friction for all types of coatings have close enough values and with loadings more than 1,0 kN they are within 0,06 to 0,1. The lowest coefficient of friction had the pair of coating *Avinit C320-n1* - coating *Avinit C220-t20/10*. The value of coefficient of the pair of friction did not exceed 0,095 in all the range of loadings, and with the maximum loading it was 0,065, that corresponds to the minimum value received in this operation for pairs of friction with tested coatings.

All coatings in tests showed high resistance against wear which value did not exceed 0,8 microns. Value of wear on samples with coating fluctuates from 0.6 to 0.8 mm.

Pairs of friction which working surfaces have micro- and nano-layer coatings *Avinit C/P 320-ms1, Avinit C/P 320-n1, Avinit C/P 350-t20/10, Avinit C/P 220-t20/10, Avinit C/P 220-t16/10, Avinit C/P 220-t16/5*, tested in the conditions of boundary lubrication, are characterized by:

- high resistance against teasers formation; absence of secondary conformability; high enough stability in timing of the coefficient of friction while operating with invariable loading; essentially smaller distinction between "direct" and "reverse" pairs, in comparison with "basic" pair *bronze VB23NS / steel 30H3VA.*

The pair of friction *Avinit C/P 220-t16/10 / Avinit C/P 320-n1* showed the best tribological characteristics in this work, namely: - friction coefficients (0,109 … 0,129) with durable operation under invariable loading are close enough to the "original" pair and are stable; - weight wear revealed after 8 hours of wear tests in "reverse" pairs, is less, than for the "original" pair a) minimum 12 times for the pair as a whole; b) minimum 2,5 times for a more solid sample of the pair; c) minimum 44 times for a softer sample of the pair;- for "direct" pairs after 8-hour tests by the used monitoring methods weight wear was not revealed.

## 5. CONCLUSION

There have been conducted metallographic and tribological researches of advanced constructions of multilayer coatings of *Avinit* type - coatings on the basis of Ti-Al-N system - *Avinit C/P 310-ms1, Avinit C/P 300-t10, Avinit C/P 100-t10, Avinit C/P 320-n1, Avinit C/P 350-t10* and coatings on the basis of Mo-N system - *Avinit C/P 210-m1* and *Avinit C/P 220-t10.* As tribological tests have shown, coating very effectively affects resistance of a pair to teasers, leading to increase in $P_{cr}$ value of teasers formation.

Tested pairs with coatings had low coefficients of friction under loadings up to 2.0 kN. Pair *Avinit C/P 320-ms1 / Avinit C/P 220-t16/10* had the least coefficient of friction. Its value did not exceed 0,095 in all the range of loadings, and under the maximum loading were 0,075. In the tests all advanced coatings have showed high wear resistance which value did not exceed 0.8 microns.

Pair *Avinit C/P 320-n1 / Avinit C/P 220-t16/10* have shown the best combination of wear resistance and tribological properties. It had the least coefficient of friction and almost zero wear during 8 hours of tests.

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