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Hi-tech technologies based on advanced fundamental research

Vacuum-plasma multilayer protective coatings for turbine blades

Monograph

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Abstract

The methods of creating the advanced nanomaterials and nanotechnologies of functional multicomponent coatings Avinit (mono- and multilayer, nanostructured, gradient) to improve the performance of materials, components and parts are considered.

The vacuum-plasma nanotechnologies Avinit were developed based on the use of gas-phase and plasma-chemical processes of atomic-ionic surface modification and the formation of nanolayer coatings in the environment of non-steady low-temperature plasma.

Considerable attention is paid to the equipment for application of functional multilayer composite coatings: an experimental-technological vacuum-plasma automated cluster Avinit, which allows to implement complex methods of coating, combined in one technological cycle. The information about the structure and service characteristics of Avinit coatings has a large place.

The results of metallographic, metallophysical, tribological investigations of properties of the created coatings and linking of their characteristics with parameters of sedimentation process are described. The possibilities of parameters processes regulation for the purpose of reception of functional materials with the set physicochemical, mechanical complex and other properties are considered.

The investigation of creating of multilayer protective surface coatings Avinit based on Ti-TiN for turbine blades by vacuum-arc method was carried out.

The influence of different methods and modes of vacuum-plasma treatment of coated surface of substrates to the adhesion value of nanolayer protective Ti-TiN coatings is studied. On the basis of carried out investigations the technology of coating the steam turbines blades for protection against flow-accelerated corrosions is developed. The issues of development and industrial implementation of the latest technologies for applying wear-resistant antifriction coatings Avinit with the use of nanotechnology to increase the life of various critical elements of steam and nuclear turbines are covered in detail.

The book is aimed at specialists working in the field of ion-plasma surface modification of materials and functional coatings application.

Keywords

Vacuum plasma multilayer protective coatings Avinit, titanium, titanium nitride, turbine blades, development of nanotechnologies Avinit.

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Preface

The task of creating new modern materials with improved performance characteristics when working in extreme conditions is closely related to the research and development of nanostructured materials and nanotechnologies.

The monograph presented to the attention of the reader presents the results of the authors' research on the development and practical implementation of the latest nanomaterials and Avinit nanotechnologies to improve the characteristics of materials.

A distinctive feature of these works is the complex use of complex vacuum-plasma coating methods (plasma-chemical CVD, vacuum-plasma PVD (vacuum arc, magnetron), ion saturation and ionic surface treatment processes) under the influence of non-equilibrium low-temperature plasma, combined in one technological cycle.

The experimental and technological equipment created for these purposes — the vacuum-plasma automated cluster Avinit — makes it possible to implement complex technologies for applying multicomponent coatings (single- and multilayer, nanostructured, gradient) for various functional purposes (antifriction, strengthening, etc.).

The monograph presents the results of the authors' work in a wide area — from fundamental research to the development of pilot technologies based on these studies and their serial implementation in turbine construction.

Circle of readers and Scope of Application

The large factual material is undoubtedly interesting and useful for engineering and technical specialists involved in the development of new materials, and will contribute to the further development of this promising direction.

Attention is paid to the prospects for the development of Avinit vacuum-plasma nanotechnologies and the expansion of applications of these methods in mechanical engineering, aviation, power engineering, astronautics and other branches of science and technology.

The theoretical and experimental results presented in the monograph are of considerable interest for scientific and technical specialists engaged in solving the problem of increasing the durability of materials with improved functional, in particular, tribological characteristics.

The book is intended for specialists working in the field of ion-plasma surface modification of materials and the application of functional coatings.

1

Introduction

The operation reliability and service life of various heat engineering equipment: turbines, steam generators, heat exchangers, valves and control valves, pumps, etc. largely depends on the degree of wear of the working surfaces of highly loaded machine elements, particularly working blades of steam turbines and compressors of gas turbine engines (GTE) and gas turbine units (GTU).

The greatest number of damage cases, that significantly reduce the service life is due to erosion, corrosion and flow-accelerated corrosion of structural materials that are in the course of operation are subjected to the simultaneous damaging effect of corrosion-erosion factors in conditions of gas-abrasive and wet-steam environment at temperatures up to 500–540 °C, a significant dynamic and static loads [1–3].

To date, the possibility of significant increasing the equipment durability by improving the properties of structural materials, the introduction of more advanced design and technological solutions are almost exhausted.

Usually, in production conditions strengthening of steam turbine working steel blades is carried out by soldering of stellite (WC) plates on the leading edge or by deposition of a very hard coating in air atmosphere (supersonic and detonation sputtering).

As it was proven by operating experience, such methods are ineffective. Soldering of the plates does not solve the erosive wear issue. At the same time, this significantly deteriorates the aerodynamic properties and efficiency factor of the turbine. The coating sputtered in the air is characterized by a weak adhesion to the substrate and low endurance due to the occurrence of oxide films.

The most effective way to improve the wear resistance of structural materials is the use of protective coatings [4, 5].

Vacuum ion-plasma coating technology has become widespread in various branches of engineering, mainly to enhance the products durability (including erosion, corrosion and fretting resistance).

In the technological aspect has an exceptionally difficult issue of the application of the required coatings on the elements having complex configuration and a large length, and depositing of coating on the parts inner surface.

Because in most cases temper of materials of the parts coated is not allowed, depositing of vacuum-plasma coatings should not be carried out at temperatures which can reduce the base hardness, heat-treated to high hardness (~65 HRC 55).

No deformations and distortions are tolerated on the coated parts. Tempering temperature of steels normally used for the manufacture of parts is 200–250 °C, therefore, the technology must provide an implementation of the low-temperature coating processes, with good adhesion to the base, at temperatures at least, not exceeding 200 °C.

Preferably no additional machining should be applied to the surface after coating. In addition, the coating must provide a significant increase in the service life of the friction pair at a thickness which does not lead to going beyond the tolerances of dimensions of parts which may be only a few micrometers, and does not reduce the treatment cleanliness class of surfaces covered, which is determined by the requirements of the design documentation, as further additional machining of parts surface after manufacture is almost impossible in most cases.

It is sufficient to only mention the enormous costs — financial and technological necessary to achieve the necessary size of parts of complex configuration and large relative length with stellite coatings.

A large number of works is known on sputtering of vacuum-plasma coatings for the protection of turbine blades [6–13].

Many researches on studying of properties of vacuum-plasma coatings concerning work of constructional materials of the thermal engineering equipment in the conditions of action of erosion corrosion and corrosion-fatigue factors are carried out.

Wear-resistant and corrosion-resistant coatings can be layers of corrosion-resistant metal layers, which are repeatedly alternating, and are selected from the group consisting of molybdenum, niobium, tantalum, tungsten, chromium, titanium, zirconium, nickel or alloys based on them.

In work [7], a three-layer coating was developed, the first layer of which consists of a layer of one metal or metals alloy of 1VA or V1A groups of the Periodic table of elements formed in a neutral gas environment, the second one — in a mixture of neutral and reaction gases, and the third one — a layer of nitrides, carbides, borides or alloys thereof.

The coating contains a layer of scandium, yttrium or rare earth metal having thickness of 0.02–0.08 μm, the number of layers can vary from 10 to 500, the ratio thickness is (0.02–5.0) : (0.04–10) : (0.1–12.5), the thicknesses of the first two layers have ration 1.0 : 2.0 : 2.5.

Tests of these coatings revealed improved corrosion and erosion resistance of titanium blades in comparison with the resistance to erosion and corrosion without coating, but low adhesive ability significantly reduces the operational reliability of the products.

Coatings resistant to erosion are described in [8]. They contain solid nitrided layer created on the surface of the core material and at least one hard layer obtained by PVD method, created on nitrided solid layer, in which layers of chromium nitrides, titanium nitrides, and nickel nitrides are created, which alternate with layers of nitrides of chromium/aluminum, titanium/aluminum, chromium and titanium. Such coatings improve anti-erosion properties of the surface, however, this does not always provide the necessary erosion and corrosion resistance of turbine blades during their operation under conditions of wet-steam erosion due to poor adhesion ability.

In [9] a method of coating in vacuum is described, comprising preliminary cleaning the product surface, in which passivation-deformation treatment is performed by a flux of high-energy neutral particles before ignition. The of result passivation-deformation treatment is obtaining a high-strength coating with high density and passivity, which increases its durability significantly. But this method is more suitable for treatment of products that do not work in environments with a considerable load of erosive factors.

A method of depositing a multilayer coating on metal products by means of cathode sputtering is also known; it comprises ion cleaning and/or surface modification of products, application of at least three-layer wear-resistant coating by sputtering of the metal layer and solid solutions of gases and layers of nitride, carbide and/or borides [10] in an inert gas environment.

The method of obtaining erosion-resistant nano-layer coating for the turbo-machines blades is described in [11]. The coating contains nano-layers that include a metal sublayer and nano-layers of titanium nitrides [11], as well as carbides and/or titanium carbonitride, zirconium carbonitride, aluminum carbonitride, and nano-layers of these metals compounds with nitrogen and carbon, as well as the implanted ions.

Vacuum-plasma application of metal sublayer and layer based titanium nitrides, which are form during the rotation of the blades relative to their own axis [11], and relative to consecutively located cathodes of different materials, in which their ion-implantation treatment is carried out after depositing of each layer.

The above-described technical solutions are used to protect turbine blades against salt and gas corrosion, gas-abrasive and drip-impact erosion. These coatings increase the blades resistance to salt corrosion and drip-impact erosion by 1.5–2 times.

In [12] erosion and corrosion-resistant ion-plasma protective coatings based on titanium nitride for blades steel protection are described.

Obtaining high quality firmly joint coatings of the multilayer structure requires the good condition of surface on which the coating is deposited, as well as layers interface surfaces. Therefore, the technological processes of the surface pre-treatment before depositing coating and in the course of coating deposition are particularly important.

The possibility of pre-treatment of the surface with high-energy ions is the most distinguishing feature of all ion-plasma technologies.

Due to the surface treatment by the high-energy particles flow it is possible to achieve modification of the materials surface properties, which usually has a positive effect on the properties of the formed coatings.

To improve the microstructure of the surface layer the new effective plasma methods of surface components preparation (plasma annealing, plasma hardening, plasma modification) is used successfully.

Electrolytic-plasma polishing of parts is broadly used [13].

In combination with surface ultrasonic treatment such combined methods of vacuum ion-plasma treatment are capable of ensuring elimination of possible deficiencies in the previous process operations to improve the cleanliness class of the surface to roughness grade 12, 0.025–0.05 R_a, which contributes to improvement of adhesion characteristics at the next deposition of vacuum-plasma coatings.

To improve operational properties of steam turbines working blades [14] multilayer nanostructured vacuum-plasma coating system of Ti-TiN system were developed using a combined vacuum ion-plasma treatment.

Processing includes pre-electrolytic-plasma surface polishing, ion-beam cleaning of the surface from oxide films, ion implantation of the surface with subsequent depositing of multilayer vacuum-plasma coating of very hard nitride compounds: Ti-TiN Zr-ZrN. All operations are carried out in one vacuum chamber. Such coatings (with thickness of 40 μm) are distinguished for strong adhesion to the blades material and high corrosion and wear resistance.

Thus, these data allow to conclude about the prospects for use in the energy of vacuum-plasma wear-resistant protective coatings that increase the life of power equipment at least to the design, and in some cases significantly increase it (3–4 times) by significantly reducing erosion, corrosion and corrosion-erosion wear of the surface of structural materials.

2

Experimental equipment. Avinit vacuum plasma cluster

It is possible to satisfy the requirements, often contradictory, to the surface properties (high hardness and wear resistance, high antifriction characteristics) and bulk properties (high strength and toughness) by creating compositions with layered arrangement of materials that perform different functions.

The most successful solution to these problems is provided by technologies for modifying the surface layers of contact materials and applying wear-resistant and antifriction coatings to improve the tribotechnical characteristics of friction pairs.

Without coatings for various functional purposes, it is impossible to imagine modern technology and further progress in all areas of its application. Increasingly high requirements for the properties of materials and the complex nature of these requirements stimulate the constant search for new materials and technologies for their production.

Coating of certain materials not only improves the properties, but leads to the creation of a new composite material with its own set of characteristics.

Among the methods of coating, a special place is occupied by methods of forming coatings from ionized atomic and molecular fluxes. The ability to change the energy of ionized particles of condensed matter streams in a wide range (from units to hundreds and thousands of electron volts) can effectively influence most important in practical application characteristics of coatings (density, adhesion, structure, etc.) and thus achieve high values of the corresponding indicators in comparison with other methods. An important feature that distinguishes these methods is the ability to create multicomponent composite materials in nonequilibrium conditions of their formation.

Vacuum coating technologies have taken a leading place in many areas of industrial production of various products of modern technology. This became possible due to the success in the development of technology for the formation of high-quality coatings for various functional purposes (protective, wear-resistant, optical and others), and the creation of appropriate industrial equipment for coating. Further progress in the application of vacuum coatings is associated with the development of technologies and equipment that would provide the ability to obtain multifunctional coatings of complex composition, high homogeneity, reproduction in composition and other characteristics.

We have created [15–17] vacuum-plasma complex (cluster) Avinit for application of multicomponent multilayer coatings by complex ion-plasma and plasma-chemical methods and testing of technological methods and industrial technologies for obtaining nanolayer composite functional coatings on parts with different geometry surfaces.

The peculiarity of the developed coating processes lies in their complexity: different coating methods (plasma chemical CVD, vacuum-plasma PVD (vacuum arc, magnetron), ion saturation and ionic surface treatment processes) are combined in a single technological cycle. The use of gas-phase and plasma-chemical methods in combination with other methods of coating and surface modification (ion doping, implantation, vacuum-plasma, diffusion, vacuum-thermal methods, etc.) significantly expands the possibilities of creating fundamentally new materials.

A wide range of coatings (almost any element, refractory oxides, carbides, nitrides, cermet compositions based on refractory metals and oxides), as well as the unique structure and properties of ion-condensed materials (amorphous, nanocrystalline, microlayer structures, etc.) open new opportunities for the creation of components and parts for various purposes to work in extreme conditions of temperature, aggressive environments, mechanical loads. The main efforts are aimed at the formation of nano- and microlayer multicomponent coatings as the most promising to achieve the required functional characteristics. Nanolayer nanocomposite coatings have great potential in creating materials with unique properties, including exceptional hardness, strength, chemical stability, low friction coefficient and high wear resistance due to the possibility of combining in various combinations of different materials and varying the thickness of the layers.

Technological vacuum-plasma automated cluster Avinit for multicomponent multilayer coatings is a high-vacuum unit with high energy saturation of power supplies of various types (gas-phase and vacuum-arc evaporators) (Fig. 2.1).

Fig. 2.1 Avinit vacuum-plasma automated cluster

It consists of such functional blocks:

- Avinit C unit for application of hard and superhard multilayer and nanolayer functional coatings by the method of modernized vacuum-arc spraying;
- Avinit V unit for gas-phase (CVD) and plasma-chemical (PECVD) coating deposition on parts internal and external surfaces;
- Avinit M unit for coating by magnetron sputtering;
- Avinit N unit for ion-plasma treatment;
- Avinit E unit for ionic etching and purification processes;
- Avinit T unit for heat and thermochemical processes.

The Avinit vacuum-plasma automated cluster is created for working off methods of receiving composite functional coverings on details with surfaces of various geometry with precision processing.

The solution of these problems is provided by the use of complex methods – CVD (gas-phase and plasma-chemical deposition) and PVD (vacuum-arc and magnetron sputtering) in combination with ion-plasma surface modification to form multicomponent multilayer nanocoatings with high functional characteristics.

It is important to use automated equipment with vacuum-arc sputtering sources with plasma filtration from the drip component, which is able to ensure the requirements for maintaining the cleanliness of surfaces with precision treatment and the appropriate degree of control over processing and coating due to the requirements for geometry and size of such surfaces.

In the presence of such capabilities of the equipment control system, it is possible to form multicomponent nanolayer coatings with the most diverse ratio of elements that are part of the layers. The ability to control the energy of ions in vacuum-plasma processes allows to influence the processes of nucleation, growth of the coating, and hence its structure, the level of internal stresses and other characteristics.

The expediency of using one or another method in the technological scheme of formation of functional coatings is primarily determined by its capabilities and features of application.

The Avinit multi-purpose complex provides a mode of operation when using any evaporating devices, systems and control devices that are part of it, while allowing the simultaneous operation of all the same type of evaporators or alternating (in any sequence) different types of evaporators. The Avinit complex is not focused on any specific technological process, but provides for the possibility of implementing various technological processes, which can use different methods of coating and pre-treatment of the substrate. It is presented in Table 2.1.

Use of effective methods of surface cleaning – in glow discharge Ar, in high-density plasma discharge and metal ions at voltage above zero point of growth, and also prevention of damage of a surface by microarcs by means of the effective three-level system of arc quenching provided in Avinit equipment, oxides and other contaminants without electrical breakdowns.

When using spray systems, coatings can be applied by igniting a DC or RF discharge, which can be excited in an inert gas, reactive gas or in multi-component gas mixtures, which include an inert gas and one or more reactive gases. Pure metals, alloys, various combinations can be used as targets that allows to receive a coating of practically any set structure in the mode of simultaneous or consecutive work of magnetron sources.

Table 2.1 Vacuum-plasma methods of coating and surface modification implemented in the vacuum-plasma cluster Avinit

Vacuum-arc coating method	Avinit C unit
Gas phase deposition methods (CVD and PECVD)	Avinit V unit
Magnetron coating method	Avinit M unit
Methods of plasma diffusion saturation	Avinit N unit
Methods of ion-plasma purification	Avinit E unit

2.1 Avinit C unit (vacuum-arc coating methods)

The installation provides the application of monolayer, multilayer, nanolayer and nanostructured coatings of metals, alloys and their compounds with nitrogen, carbon, oxygen.The presence of an effective system of protection against arc and microarc discharges, which can damage the surface of the coated parts, as well as plasma separation devices from the drip component of the arc evaporators ensure the preservation of surface finish at a level not worse than 11–12 class. This allows to apply coatings on precision surfaces of high purity, eliminating the need for any finishing coatings after application.

Gas supply to the chamber of inert and reaction gases is carried out by 2 independent lines with regulators – flow meters of fine regulation from 0 to 200 cm^3/min of the Bronkhorst company (Holland).

2.2 Avinit V unit (gas phase deposition methods (CVD and PECVD)) [16]

The Avinit V unit for vacuum-plasma and plasma-chemical surface treatment and application of functional coatings is designed for experimental development of new technologies for the application of metal and metal-carbide coatings based on molybdenum and tungsten by decomposition of their carbonyls.

The Avinit V unit is designed for the implementation of coating processes by thermal decomposition of organometallic compounds, mainly hexacarbonyls of metals VI-B of the Mo, W, Cr groups and their compounds with nitrogen, carbon, and others.

The use of gas-phase and plasma-chemical methods in combination with other methods of coating and surface modification (ion doping, implantation, vacuum-plasma, diffusion, vacuum-thermal methods, etc.) further expands the possibilities of creating fundamentally new materials and coatings.

2.3 Avinit M unit for coating by magnetron sputtering

The Avinit vacuum-plasma cluster provides for the possibility of using various sources of sputtering, in particular magnetron sputtering systems.

The Avinit M unit is intended for magnetron coating and experimental testing of new technologies for coating of and non-conductive materials of HF and DC magnetron sputtering of targets.

The magnetron is powered by a DC power supply with a strongly decreasing volt-ampere characteristic with a power of 2 kW and an idle voltage of 6 kV.

The RF power is supplied to the inductor through a matching device from the serial generator UV-1 with an operating frequency of 13.56 MHz and a power of 1 kW.

The Avinit M unit is equipped with a modern high-voltage power supply of the magnetron and devices of plasma and plasma chemical surface treatment of TruPlasma DC01 BP Kurt J. Lesker products with a capacity of 1 kW with output voltage up to 800 V.

The source provides operation in stabilization modes for power, current or voltage with protection from overloads at interruptions of burning of plasma of a glowing discharge in a microarc or arc modes, and also provides a possibility of remote computer control.

The Avinit M unit provides an opportunity to put coverings from metals and their various connections, including, not electrically conductive, and also nonmetals (carbon, fluoroplastic, optical, etc.) and different in composition and structure (monolayer, multilayer, including nanolayer and nanostructured).

This variety of coatings makes the method of RF magnetron sputtering of targets and technologies based on its use, one of the most popular in a number of areas of modern production, as well as in the development of new types of coatings and technologies for their production.

The Avinit M unit with magnetrons (DC and RF) is fully combined with the Avinit N unit and the Avinit C unit, which allows the development of hybrid technologies.

2.4 Avinit E unit (ionic treatment (cleaning) of the surface before coating)

The Avinit E unit, designed for ion-plasma treatment of materials, is equipped with devices for the implementation of plasma glow discharge of high density, excited in the hollow cathode.

This allows the development of new and improvement of existing technologies of ion-plasma surface treatment of materials in the plasma of the glow discharge.

The installation provides the ability to process the surface of materials in plasma glow discharge of high density in gaseous media of different composition (argon, nitrogen, gases containing carbon, and mixtures of these gases), including internal cavities and channels of certain sizes. The use of high-density glow discharge plasma in the processes of ionic nitriding, nitrocementation can reduce many times compared to gas furnace technologies the time of formation of reinforced layers up to 0.3–0.4 mm, while maintaining virtually unchanged dimensions of the processed products and preventing the formation of brittle phases on their surface. Therefore, the development of new technologies for surface treatment in high-density plasma is one of the current areas of improvement of modern technologies in a number of industries.

Structurally, the Avinit E Unit can be combined with the Avinit C Unit and the Avinit N Unit for glow discharge cleaning, metal ion cleaning in a vacuum arc discharge, and high-density double-arc plasma cleaning in high-density plasma.

Ion-plasma methods provide a high value of adhesion of coatings due to the high energy of condensing ions and pre-cleaning of the coated surface.

Ionic treatment of products can be carried out using special ion sources or in plasma gas discharge, which is ignited in the volume of the vacuum chamber.

The basis of ion-plasma methods of surface cleaning are the processes of interaction of vacuum-plasma flows with the surface of the substrate material.

The nature of the interaction of particle fluxes falling on the surface with the substrate material is determined by the flux density of particles, their energy, degree of ionization, temperature in the interaction zone and may consist either exclusively in spraying, etching of surface layers (at high ion energies) or condensation (at low energies).

The consequence of the purification process (ion bombardment) is:

- purification from sorbed surface atoms of contaminants;
- heating of the substrate material;
- degassing;
- selective etching, which changes the morphology (purity of processing of the surface;

- activation of surface atoms, which ensures the flow of plasma-chemical processes of interaction with the atoms of condensed matter.

All these factors contribute to the formation of high adhesive bonds of the coatings to the substrate material.

In the Avinit installation, the surface can be treated with inert or reaction gas ions (Ar, N_2, etc.) when the glow discharge is ignited.

Due to the lower ion density in the glow discharge plasma, the intensity of heating and the associated gas evolution from the surface to be covered will be less and the intensive formation of microarc discharges can be avoided.

Devices with electric arc evaporators, in addition to the known advantages, have a number of serious disadvantages.

First of all, this applies to problems arising from ionic cleaning in electric arc spraying due to the appearance of erosion traces from the cathode spots of the arc discharge (microarcs), which leads to a significant deterioration in the purity of the treated surface, as well as a significant number of macroparticles ("drip component"), which are generated by the cathode spot of the vacuum arc.

There are technological problems in which the presence of defects in the coating caused by metal microparticles is absolutely unacceptable (application of functional coatings on precision surfaces, anti-corrosion, decorative, optical, etc. coatings).

In installations with electric arc evaporators, the working volume is filled with highly ionized metal-gas plasma.

Effective ionic purification is possible if there is highly ionized gas plasma in the working volume.

In the Avinit installation taking into account complexity of methods (plasma-chemical, vacuum-arc, magnetron) and also specifics of geometry and precision of a surface of the covered products, for replacement of metal-gas plasma by gas plasma the gas plasma generator on the basis of two-stage vacuum gas plasma for the purposes of highly efficient ionic treatment by cathodic spraying of the surface with ions of the working gas, which provides a strong adhesion of the coating to the substrate and the application of high quality functional coatings.

When ion-plasma purification using DVDR in the presence of gas plasma there is no problem of deposition on the surface of metal particles, and therefore the potentials for the product can be changed smoothly, starting from zero. It is possible to achieve the complete absence of electrical breakdowns on contaminated areas of the surface in comparison with the case when the complete cleaning of the surface is carried out by metal ions, and thus achieve the preservation of the original purity of the treated surface.

In addition, purification by gas rather than metal ions may have the advantage that often the gas ion sputtering ratio is higher than the metal ion sputtering ratio, and therefore the ionic purification process at the same ionic current and ion energy is more intense.

And although the power consumption in a DVDR installation is twice the power consumption in a magnetron installation, which makes the use of magnetrons for cleaning purposes more common, the possibility of efficient surface cleaning of complex products with high surface finish (eg hemispheres) using DVDR when spray targets can be of almost any shape, and the use of magnetrons is inefficient, the use of two-stage arc discharge (DVDR) is very appropriate.

2.5 Avinit N unit (ion-plasma surface modification) [17]

The Avinit N unit is intended for carrying out processes of ion-plasma modification of surfaces, in particular, for ion-plasma surface treatment, plasma diffusion saturation (nitriding, nitrocementation, etc.) of steel and alloy parts in high-density low-temperature nonequilibrium plasma, in precision, without processes of diffusion saturation in plasma with a poly cathode.

The Avinit N unit is designed in such a way that it is possible to connect devices for simultaneous magnetron sputtering processes.

The Avinit N unit is equipped with a gas plasma generator for carrying out processes of vacuum-plasma high-precision nitriding of steels and alloys in high-density low-temperature nonequilibrium plasma.

Plasma burns evenly in large volumes, providing uniform heating of details to necessary temperature and nitriding of difficult-profile products of various forms and the sizes, including through and deaf openings. Its density is several orders of magnitude higher than that of ionic nitriding in a conventional glow discharge, as a result of which the process of formation of the nitrided layer intensifies 2–5 times in comparison with the traditional method of ionic nitriding in a glow discharge and 5–10 times in comparison with gas nitriding. This ensures the absence of deformation (curvature) of the parts while maintaining the original geometric dimensions after nitriding with an accuracy of 1–2 μm, there is no brittle surface layer, typical of traditional methods of nitriding. This avoids finishing grinding of parts and to carry out the operation of precision nitriding "in size".

2.6 Power supplies of the Avinit installation

The ionization degree of the vapors of the evaporating substance depends on the parameters of the evaporator, the composition of the evaporating substance, and the conditions of excitation of the discharge, the design features of the nodes, the ionization system of vapors.

Methods of vapor ionization using an arc discharge require high-current (tens and hundreds of amperes) power supplies with a voltage of up to

100 V, which are equipped with all serial installations of vacuum-plasma spraying. The Avinit installation also includes the use of plasma sources, which are equipped with magnetron sources of DC and RF current.

When using glow discharge technology schemes, the most effective methods are those that use a non-self-contained glow discharge. The presence of power supplies of magnetron evaporators on direct current as a part of Avinit installation allows to provide realization of schemes of not independent glow discharge with use of direct-burning cathodes without essential additional expenses.

When the glow discharge is excited, the RF discharge has certain advantages over the DC discharge. It can be excited at a lower electric field strength and lower pressure, provides a higher density of ions in the discharge, can be implemented in the "non-contact" version.

Power supplies of the RF-magnetron evaporators, which are part of the Avinit unit, are used to power the RF discharge system, providing in them the possibility of coordination with the inductive load.

The existing in-cluster system of inert and reaction gases in the Avinit cluster is able to fully provide the necessary conditions for the pressure of the medium to excite the discharge in the vapor ionization circuit of the evaporator at almost any mode of evaporator operation, including operation at minimum power. CVD processes using non-equilibrium low-temperature plasma are implemented in the Avinit installation. Low-temperature non-equilibrium plasma of low pressure in the environment of the used precursors is ignited by means of standard RF generators and metal concentrators specially designed according to the geometry of the vacuum chamber.

The plasmaization possibility of the gaseous medium in the implementation of CVD-processes significantly expands the possibilities of implementing the latest methods and technologies of coating in the Avinit installation. One of the most interesting directions in development of PECVD methods is the method developing now connected with use of plasma of high density (HDP-CVD). The generated ion density for this method is from 10^{11} to 10^{13} ions/cm^3, while for conventional PECVD methods it is in the range of 10^8...10^{10} ions/cm^3. Unlike conventional PECVD processes, electromagnetic energy in HDP-CVD processes is transmitted in a relatively small spatial region of the reaction volume, and the plasma flow from the excitation zone is directed to the substrate, which is outside the electromagnetic field that excites the plasma.

Due to the selected design of the chamber, the configuration of the magnetic field, the elements forming the gas flow, a uniform flow of plasma to the substrate is created. And although the plasma density is reduced by about 10 times compared to the excitation site, it is possible to change the plasma density in a wide range without affecting the deposition zone of the coating by the exciting electromagnetic field, which makes the process more controlled and controlled.

2.7 Measurement of plasma source parameters of the Avinit cluster

The solution to the problem of obtaining multifunctional coatings with the required characteristics is largely related to the possibility of more complete control of technological parameters and their support and management in automatic mode. First of all, it concerns plasma parameters.

In the Avinit installation realization of the following plasma sources used for the purposes of drawing functional coverings and surface modification is possible:

- glow discharge plasma (surface cleaning and ion saturation);
- plasma vacuum-arc discharge (reactive coating);
- magnetron discharge plasma (reactive coating);
- plasma double arc discharge (surface cleaning and ion saturation);
- HF plasma for the implementation of deposition processes from the gas phase PECVD, activated by low-temperature nonequilibrium plasma (reactive coating).

In [18, 24] a comparative analysis is done for different plasma sources used in reactive coating and diffusion saturation of metals in the Avinit installation.

To measure the plasma parameters, the Langmuir probe method was used, implemented using the PlasmaMeter device. A tungsten probe with a diameter of 0.1 mm and a length of 5 mm with an L-type support assembled from metal and ceramic tubes was introduced into the vacuum chamber through a movable seal, which allowed 2-coordinate movement of the probe inside the chamber without violating vacuum conditions (Fig. 2.2).

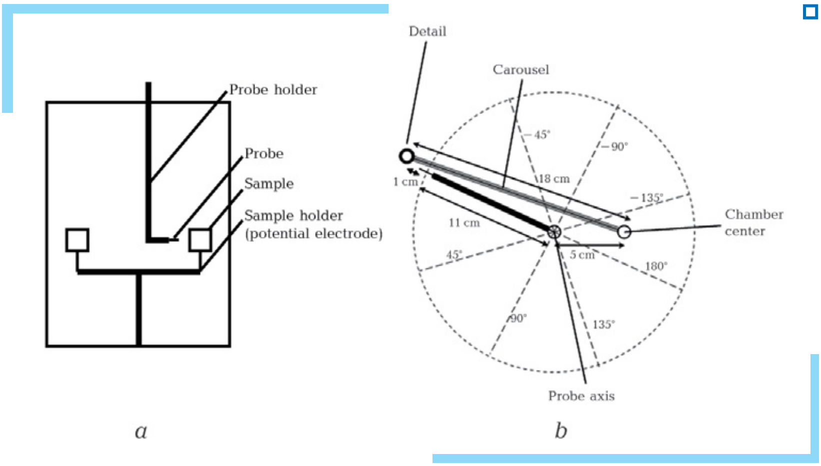


Fig. 2.2 Location of the Langmuir probe in the vacuum chamber of the Avinit installation: *a* — cross-sectional side view; *b* — a top view

Probe measurements of process plasma parameters in the Avinit installation were performed [18] using fundamentally different sources that cover a very wide range of plasma parameters.

In [18], the results of measurements of the parameters of two plasma sources intended for coating (magnetron and arc) and two gas discharges for diffusion saturation of metals (incandescent and double arc) are presented.

Experimental study of different plasma sources of the Avinit installation showed that the achievable plasma parameters cover a wide range, which provides an opportunity to implement a wide range of technological processes.

Table 2.2 summarizes the results of measuring the plasma parameters of all four sources used in Avinit installations.

Table 2.2 The results of measuring the parameters of plasma sources

Type of discharge	Plasma density, cm^{-3}	Electron temperature, eV	The degree of ionization
Magnetron	10^{10}	5	$3 \cdot 10^{-4}$
Arc	$4 \cdot 10^{9}$	0.4	0.5
Smoldering	10^{8}	0.15	10^{-7}
Double arc	$6 \cdot 10^{9}$	5	10^{-4}

The results of the glow discharge study show low values of plasma density and ionic current density on the treated surface. Such plasma can be successfully used for surface cleaning or ionic assistance in coating, however, the technology of diffusion saturation of metals (eg, nitriding) requires the use of denser plasma. The Avinit installation uses a double arc discharge for this purpose, which allows you to create a high-density plasma in the chamber. Estimation of the ratio of ion fluxes and neutral atoms for plasma of a double arc discharge gives a value of the order of 10^4 atoms per ion, ie approximately 300–1000 times more intense ion flux compared to the case of the glow discharge. Such plasma is successfully used in the Avinit installation both for cleaning of a surface or ionic assistance at drawing functional coverings, and in technologies of diffusion saturation of metals (for example, nitriding) with use of much denser plasma.

An important role in plasma technology is played by the homogeneity of the plasma in the volume of the chamber, because in group processing to comply with the optimal technological regime, all machined parts must be in the same conditions. To study the homogeneity of the plasma of the double arc discharge, the plasma parameters were measured at seven angular positions of the probe at three levels along the height of the chamber, at a distance of 170, 300 and 470 mm from the bottom wall of the chamber. The measurement results showed high homogeneity (±10 %) of plasma throughout the chamber. This result is extremely important for increasing the stability of technological operations to obtain high quality products for coating and modification of metal surfaces.

3

Experimental studies of Avinit coating processes

3.1 The study of the structure and tribological properties of Avinit coatings

3.1.1 Method of coating

The development of processes for the application of new functional multilayer composite coatings was carried out on a vacuum cluster Avinit, created for the implementation of complex coating methods (plasma chemical CVD, vacuum-plasma PVD (vacuum arc, magnetron), ion saturation and ionic surface treatment). The coating was applied according to a given program using one-component cathodes in the reaction gas medium and without it. Control of all basic parameters of the coating process was carried out automatically.

Avinit coatings were deposited on precision surfaces of high purity class up to class 12–13 without reducing the surface purity class. This was achieved by using in the developed technologies effective methods of surface cleaning — cleaning in a glow discharge Ar, cleaning in a two-stage vacuum-arc discharge and cleaning with metal ions at voltages above zero point of growth, as well as preventing surface damage by microarcs, arc extinguishing system that provides high quality surface cleaning from oxides and other contaminants without electrical breakdowns. The deposition was carried out at low temperatures not exceeding the tempering temperatures of the base, ensuring the preservation of mechanical characteristics and the absence of curvature of the coated products.

The magnitude of the current of the vacuum arc discharge with a molybdenum cathode was 140–150 A, respectively, with a titanium or aluminum cathode 100–110 A. When coating in a nitrogen environment, its pressure was in the range $(1.3–3)\cdot10^{-1}$ Pa.

An RF inductor with an operating frequency of 3 MHz and an effective power of ~0.2 kW was used to heat the sample. The temperature of the sample is monitored by an IR pyrometer through a special window in the camera door.

3.1.2 Methods of studying the properties of experimental samples

The main attention was focused on the study of the reproduction of the composition of ceramic compositions in their formation by vacuum-plasma methods, adhesion of film materials, structural state and some other properties. The degree of reproduction of the film composition was evaluated according to X-ray (X-ray diffractometer DRON-3, filtered Cu-Kα radiation) and spectral (ISP-30 spectrograph) studies, comparing the spectra of starting materials (targets) and condensed films. The study of surface morphology, fractographic studies were performed using the methods of electron, scanning and optical microscopy.

Metallographic studies (structure and properties of working surfaces (micro section, hardness of the coating, determination of surface geometry after coating) and determination of material parameters (layer thickness, uniformity, defect and structure of the material) were performed using metallographic methods (Tesa Visio 300 gL microscope), chemical, X-ray diffraction and micro-X-ray spectral analyzes, measurement of microhardness, roughness of friction surfaces. automatic mode on the device model "EKOMET 3 + AUTOMET 2" company "BUEHLER".

The microhardness of the layers was measured using a microhardness tester AMN-43 company "LECO", in automatic mode at a load of 50 G.

Measurements of microhardness and Young's modulus in multilayer and nanolayer coatings of Avinit type with a thickness of 1...3 μm were performed using a nanohardness measuring device from CSM (Switzerland) (loading speed 20.00 mH/min, max depth 100.00 nm at a load of 0.6 G, processing the results in the model Oliver-Farah).

Metal-physical measurements of the obtained coatings on mock-ups were performed on a JSM T-300 scanning electron microscope.

Measurement of the characteristics of the geometric dimensions of the control samples was performed with an accuracy of 0.5 μm before and after nitriding.

Measurement of involute surfaces was performed on a control and measuring machine Wenzel LH65 using surface points applied to the 3-D model of the part.

The surface roughness of the samples before and after coating was measured on a profilier-profilograph Jenoptik.

Removal of profiles of change of chemical individuality and nanolayers of functional coverings was carried out by means of a method of mass spectrometry of secondary ions (MSVI) on the secondary emission mass spectrometer MS 7201M. The maximum depth of profiling is 5 microns. A beam of Ar^+ ions with an energy of 5–7 keV was used for sputtering.

Examination of the functional areas of the surface of the samples was performed using scanning electron microscopy (SEM).

Removal of the volume distribution of chemical elements was performed using electron-probe X-ray microanalysis (EZRMA).

Plasma parameters (ion current, ion density, volt-ampere characteristics, spectral characteristics) were continuously monitored and archived using a plasmameter "PlasmaMeter" and a spectrometer "PlasmaSpectr".

3.1.3 Methods of research of friction and wear characteristics

Tribological tests of coated samples were performed on friction machines to determine critical values of load parameters that lead to burr, i.e. finding the limits of application of the studied friction pairs, determining the coefficients of friction and wear and their change during friction to predict material compatibility.

Tribological tests of antifriction, wear properties and setting of samples with coatings were performed on a friction machine 2070 SMT-1 according to the schemes – "cube (basic sample) – roller (counterbody)" and "ring – ring" (for wear tests) at step load in load intervals 1–20 MPa.

To determine the setting of the surface layers of the materials of the friction pairs, the load was carried out from $P_{\min}$ to the critical mark P_{cr}, at which setting takes place.

In the process of tribological tests recorded the values of friction force F_{fr}, normal load N, contact pressure P, the value of which was judged on the mechanical losses in the tribosystems. The coefficients of friction were defined as $f = F_{\mathrm{fr}}/N$.

The roughness and geometry of the friction surfaces of the samples after coating met the requirements for parts of fuel pumps.

Additionally, the wear rate was measured by the method of acoustic emission. As an informative parameter of acoustic emission used the acoustic emission power, which was estimated in relative units. The application of the acoustic emission method is an effective tool for accelerated testing, as it is extremely sensitive when registering the transition of tribosystems from normal (mechanochemical) wear to the initial destruction of the fatigue surface.

3.2 Properties of functional multilayer Avinit coatings

Properties of coatings produced by vacuum-arc deposition method depend on many parameters, and determining the optimal one in each specific case requires a significant study. We have studied the impact of the main parameters on the properties changing of the coatings based on molybdenum, aluminium, zirconium, and their compounds in the form of nitrides, carbides, oxides.

Temperature of the coating forming is an essential parameter. In many cases, coating deposition process is required to preserve mechanical properties of the basis material that can be obtained by relevant heat treatment modes, wherein tempering temperatures do not exceed 180–240 °C. This imposes certain restrictions on both coating deposition temperature, and given materials. Achievement of sufficient level of coating adhesion under given temperatures is not an easy task even for vacuum-plasma methods that compare favorably with the other types in this relation. This task requires thorough preparation and selection of the surface vacuum-plasma treatment modes, and subsequent coating deposition modes. This moment was chosen as one of the initial factors when developing coating deposition modes.

As shown by previous studies, when depositing coatings produced by vacuum-arc method in various modes, uniformity degree of the coating allocation is very sensitive to the parameters of coating deposition process. These modes vary in previous ion-plasma cleaning time, and in the value of negative offset voltage that applies to the sample in the process of coating deposition. By selecting optimal parameters of the process, it becomes possible to form coatings on the sharp edges and spherical surfaces. Besides, sensitivity of coating deposition uniformity to the process conditions makes it useful to optimize the coating process on the samples coating stage.

Previous studies are underlying factors of the time-temperature parameters selection when producing hardening coatings, applied for enhancing wear-resistance of precision friction pairs working surfaces.

3.2.1 Avinit C 100 coatings (based on titanium nitrides)

For deposition of monolayer coatings of one compound and multilayer coatings, comprising the sequence of soft and hard layers, the following technological schemes were used [19]:

a) monocathodic scheme with continuously working coating source and impulse (intermittent) reaction gas supply was performed in two versions. The first version had a substrate rotating on its axis thus covering the whole sample surface. In the second version, the substrate was not rotating having only one side of the sample coated.

These schemes were applied to produce coatings in TiN, MoN, CrN systems, and in (TiN-Ti), (MoN-Mo) systems.

b) bicathodic scheme ($h_i > \theta_i$) with two turned towards each other coating sources, working in impulse mode, and impulse supply of nitrogen reaction gas synchronized by timing with coating source work. Thus, rotating the sample on its axis allows for a hard layer forming.

These scheme was applied to produce coatings in (MoN-Cu) system.

For deposition of multilayer coatings of hard compounds, the following two technological schemes were used:

a) dicathodic scheme ($h_i > \theta_i$) with two turned towards each other coating supplies working simultaneously in nitrogen reaction gas environment, while rotating the sample on its axis;

b) dicathodic scheme ($h_i > \theta_i$) with two turned towards each other coating supplies working in impulse mode in nitrogen reaction gas environment, while rotating the sample on its axis.

These schemes were applied to produce nanostructure coatings in (TiN-AlN) system.

Studying of nitride-titanium vacuum-arc coatings deposition process has been conducted to determine optimal parameters of high-quality coatings production process using specific technological equipment Avinit. This data is essential for further producing new types of functional composite multilayer coatings in order to enhance wear-resistance of precision friction pairs working surfaces.

One of the essential coating characteristics is its composition. Studying the phase composition of coatings obtained through the methods of ion-plasma deposition of titanium in nitrogen environment, shows existence of three phases depending on nitrogen pressure. These are α-Ti, ε-Ti_2N with tetragonal crystal lattice and δ-TiN with cubic crystal lattice [20–22]. These phases have rather wide concentration areas of solid solutions and TiN compounds homogeneity. Having several phases in the Ti-N system state figure, rather wide concentration areas of solid solutions, TiN compound homogeneity, lead to differences in coatings' mechanical characteristics depending on the nitrogen concentration in them.

Nitrogen partial pressure value under which one of these phases or their set is obtained depends on both the equipment and its characteristics, and conditions of forming the coating on the substrate. Among these conditions, temperature and offset voltage are the factors of greatest impact. But despite all the differences in coatings' reaction deposition in nitrogen environment, partial pressure value under which titanium nitride phases can already be detected is at the rate close to $1 \cdot 10^{-3}$ Pa.

Increasing nitrogen partial pressure is accompanied by transition from the heterophasic coating composition (α-Ti, ε-Ti_2N, δ-TiN) with sequentially disappearing phases α-Ti, ε-Ti_2N to the single-phase state δ-TiN, wherein transition to the single-phase TiN coating can occur while nitrogen total concentration in it has already reached ~38–40 % [20, 21], that is close to the δ-TiN homogeneity limit. Crystal lattice parameter a for the massive titanium nitride has a value of 0.424 nm while films generally have a of slightly higher value (0.425–0.428) nm. The value of crystal lattice parameter a increases with the nitrogen level increasing, coating thickness declining, internal tensions increasing [20–22].

In the work [22] there has been studied possibility of forecasting the phase composition of coatings based on titanium and chrome nitrides by thermodynamic calculations of TiN system equilibrium. Calculations were conducted using entropy maximum principle [23] depending on nitrogen pressure, coatings forming temperature, coating growth rate, and the ratio of nitrogen ion flow to metal ion flow. The result of phase composition comparison based on adopted model with experimental results and data of other authors' studies, showed satisfactory results.

Depending on the nitrogen pressure while condensation, coatings can be conditionally subdivided into two groups: I — coatings of heterophasic composition; II — coatings of almost single-phase composition of δ-TiN compound (disregarding titanium-phase drops available in condensates, obtained under any nitrogen pressure value).

In condensates deposited under the nitrogen pressure lying within the range of $2 \cdot 10^{-3}$–$2 \cdot 10^{-2}$ mm Hg, nitrogen level increases from 42 at. % to 52 at. %. This is consistent with changing the ratio of nitrogen level to titanium level from 0.7 to 1.04. These values characterize TiN_x homogeneity area wherein microhardness changes monotonously with the growth of (x), reaching its maximum $H = 2 \cdot 10^4$ MPa, which is common for stoichiometric TiN. In this case, changing of the ratio of microhardness to nitrogen level occurs not monotonously, reaching the maximum value $H = 35 \cdot 10^3$ MPa under $C = 45$ at. %. With the approach of the nitrogen level in the coating to 50 % at. ($P = (2...5) \cdot 10^{-3}$ mm Hg) microhardness nears to $24 \cdot 10^3$ MPa.

With the further increasing of nitrogen pressure ($P = 5 \cdot 10^{-3}$ mm Hg) its level in the coating reaches (50...52) at. % and microhardness increases again.

In condensates obtained under $P = 2 \cdot 10^{-3}$ mm Hg nitrogen level decreases through the reducing efficiency of the nitrogen synthesis process. Consequences include increasing microdensity and decreasing microhardness. Ion energy impact on the condensates' properties has been studied on the samples of DIN 1.2379 steel. Condensation process was carried out under the nitrogen pressure of $P_1 = 6 \cdot 10^{-3}$ mm Hg, $P_2 = 1.5 \cdot 10^{-3}$ mm Hg and accelerating potential values from –50 V to –300 V, while temperature of the samples was changing from 200 °C to 600 °C.

Such changes in condensation conditions almost do not affect the microhardness value of obtained heterophasic ($P = 6 \cdot 10^{-3}$ mm Hg) and single-phase ($P = 1.5 \cdot 10^{-3}$ mm Hg) coatings. Condensation modes, especially temperature and condensation time, essentially affect the state of substrate's material. In almost all modes ensuring to obtain quality coatings tempering of DIN 1.2379 steel occurs. Thus, the higher the ion energy is while condensing, and the longer the condensation time is while the sample rotates on its axis in order to obtain coatings with uniform thickness on both sides of the sample, the stronger the steel tempers.

As studies have shown, forming Ti-based compounds require nitrogen pressure to be within the range of $3...4 \cdot 10^{-1}$ Pa.

Further studies of coating forming processes of various compositions were conducted in conditions that did not lead to the samples' temperature rise over 200 °C. On the vacuum-plasma cleaning stage it was achieved through the use of treatment impulse mode, through selecting the ratio between work and pause intervals of the arc supplies, and selecting the total treatment time. While forming coatings with the use of titanium or aluminum cathodes, as studies have shown, the close-to-optimal arc source mode involved 2 seconds of work and 4 seconds of pause with the total treatment time of 3–5 minutes and smoothly increasing accelerating potential from 30...50 V to the maximum value of 1000 V. While working with molybdenum cathode the pause time was increased to 6 seconds. On the stage of depositing coatings based on titanium and compounds with nitrogen, maintaining temperature within the limits of 180–200 °C was possible in the mode of continuously working vacuum-arc source with the potential of 30–40 V.

There is a rather high correlation between changing of coatings' phase composition and microhardness curve changing [20–22]. Despite the differences in microhardness absolute values provided by different authors, microhardness maximum (~28÷32 GPa) is in nitrogen concentration area that is consistent with the heterophasic coating composition (ε-Ti_2N + δ-TiN). It is these coatings, which show maximum wear-resistance in dry-friction conditions, where the main reason of contacting surfaces destruction is abrasive wear [24–30]. But generally, coatings' resistance under various operating conditions of the instruments, machine parts is not only defined by its microhardness value, but has a more complicated dependence on its composition, and also requires structural, orientation and other characteristics that determine coatings' physical and mechanical properties in general, to be taken into account [26–30]. Coatings obtained by titanium plasma flow deposition in vacuum $P = 10^{-3}$ mm Hg have microhardness of $4 \cdot 10^3$ MPa, that is superior to the respective values for cast material due to fine dispersed structure and the impact of residual gases' impurities. Titanium condensation under $P = 7 \cdot 10^{-3}$ Pa leads to the forming of nitrogen solid solutions in titanium with microhardness of $H = 10^4$ MPa.

One of the multilayer coatings' indicators that determine its properties in many aspects is the single layer thickness. When forming coating the necessary layer thickness is determined by working time of the relevant source that requires growth rate to be known. Generally, the coating growth rate depends on the power of the coating deposition source, on the distance from the source to substrate, on its orientation and position in relation to the axis of the diagram of coating source's atomic flow direction, on the shape of the direction diagram itself, and on the offset potential applied to the substrate. The substrate can be fixed, rotating on its axis, or be planetary rotating.

Table 3.1 shows the results of experiments to determine the growth rate of coatings.

Table 3.1 Growth rate of coatings with various composition

No.	Coating	The growth rate, V, µm/hour	Notes
1	Avinit C/P 100 (TiN)	0.9	Fixed position
2	Avinit C/P 100 (TiN)	0.25	Planetary rotation
3	Avinit C/P 110 (TiN)	0.16	-----x-----

Based on the coatings growth rate data the Avinit equipment was programmed for producing nanolayer Ti-TiN coatings with the recurrence interval of 10 nm and single nanolayers' thickness of 2 and 8 nm respectively.

These processes' automated control system protocols are described in Fig. 3.1.

Composition and some of the characteristics of the coatings studied by hardness, microhardness and roughness are described in Table 3.2.

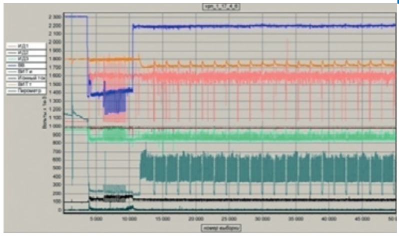


Fig. 3.1 The fragment of the automated control system's protocol of the Ti-TiN nanocoating deposition technological process with the recurrence interval of 10 nm and single nanolayers' thickness of 2 and 8 nm

Table 3.2 Characteristics of the samples

No.	The coating composition	Substrate hardness, HRC	Technological parameters			Properties of coatings		
			Programmable composition	T, °C	Nitrogen pressure P, Pa	Coating thickness, μm	Coating microhardness, HV, MPa	Roughness, R_a, μm
Ti-N – based coatings								
1	Avinit C/P 100 (TiN)	59–60	Monolayer without separator	250	$1.5 \cdot 10^{-1}$	12.0	15000–18000	0.70 (7c)
2	Avinit C/P 100 (TiN)	59–60	Monolayer with separator	250	$1.5 \cdot 10^{-1}$	1.0	15000–19000	0.040 (11c)
3	Avinit C/P 110 (TiN)	59–60	Monolayer with the recurrence interval of 10 nm and single nanolayers' thickness of 2 and 8 nm	250	$1.5 \cdot 10^{-1}$	1.0	13000–18000	0.036 (12a)

Metallographic studies of the samples with coatings of various compositions showed that developed modes had ensured forming of the quality coatings.

In the chosen modes, hardness and microhardness of the substrate material almost do not reduce compared to the initial state.

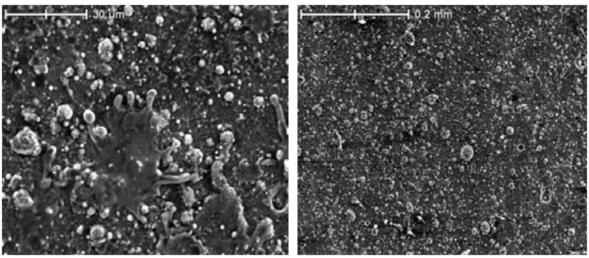
Coatings had strong adhesion to the substrate material. Exposing samples to the scratch mesh did not reveal any coating detaches.

The surface morphology of the coatings obtained through condensation from unseparated plasma flows is characterized by availability of the macroparticles (mostly metal drops), which quantity, size, and form depend on condensation physical and technological parameters.

Studying roughness of the coated samples showed that its value is determined by both roughness of the initial surface value and coatings' deposition modes.

While processing the initial surface to the 6–7 finish class coating deposition did not reduce surface finish class and depending on the deposition mode could even enhance it by several points. Thus, after depositing coatings on initial surface of the 7 class surface finish steel sample in the mode of spherical structure formation, roughness value R_a was consistent with surface finish of 10 class.

Without using the rectilinear separating device the coating surface finish, as profilographic studies have shown, deteriorates significantly (Fig. 3.2). Consequently, numerous macroparticles, common for condensation from unseparated plasma flows, emerge on the coating surface (mostly metal drops that depending on their formation time are covered with subsequent layers). Their quantity, size and form depend on technological parameters of the deposition process.



a *b*

Fig. 3.2 TiN coating (×200): *a* – without separator; *b* – with a rectilinear separator

In TiN coating obtained by usual deposition without separator initial surface roughness (class 12c) reduces sharply (class 7c, Table 3.1 paragraph 1).

After depositing coatings on the samples with roughness of 12–13 surface finish class using a rectilinear separating device, surface roughness almost does not change or little roughness increasing can be seen that almost does not exceed the limits of one surface finish class, according to the surface roughness classification.

While condensing, the heat load reduction due to decreasing ion current density, the usage of the rectilinear separator to get rid of drop phase, enhances the uniformity of condensate structure and ensure preservation of the surface finish class at the level of V13 (Fig. 3.3).

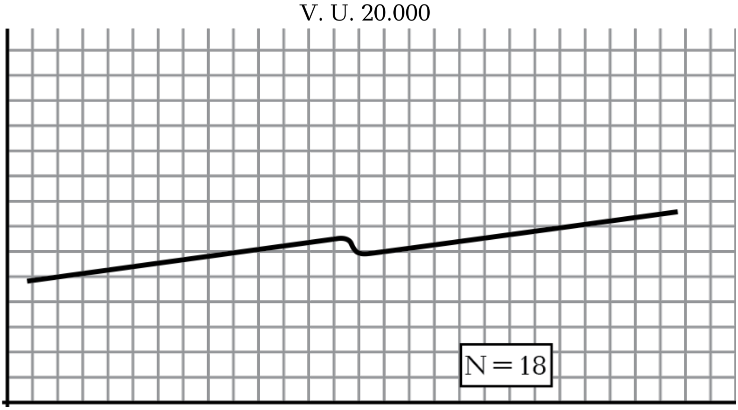


Fig. 3.3 Profilogram of the nanocomposite Ti-TiN coating

3.2.2 Avinit C 200 coating (based on molybdenum nitrides)

According to the state diagram in the Mo-N system at temperatures below 350 °C, depending on the nitrogen content, the α-Mo, α-Mo + β-Mo_2N, β-Mo_2N, and δ-MoN phases can be observed [31]. The β-Mo_2N phase has a tetragonal crystal lattice with the parameter $a = 4.2$ Å, $c = 8.01$ Å, and the δ-MoN phase has a hexagonal crystal lattice with the parameter $a = 5.72$ Å, $c = 5.608$ Å. However, as evidenced by the results of many works, the vacuum-arc coating of coatings shows the presence instead of the phase β-Mo_2N more high-temperature modification of γ-Mo_2N, which has a friction lattice with parameters $a = 4.15...4.23$ Å [31–34].

According to the work of [32] during vacuum-arc coating of molybdenum cathode in an air atmosphere at a pressure above $1.2{\cdot}10^{-2}$ Pa in the coating near the phase α-Mo is observed phase γ-Mo_2N. The amount of the latter increases with increasing pressure of the gas mixture in the chamber and at a pressure of more than $9.3{\cdot}10^{-2}$ Pa and up to the maximum pressure in these studies $1.33{\cdot}10^{-1}$ Pa coating has a single-phase structure of γ-Mo_2N with lattice parameter $a = 4.204$ Å and a microhardness of 29–30 GPa. In [33], when studying the dependence of the coating composition on nitrogen pressure, it was found that at pressures greater than 1 Pa and substrate voltages below 150 V, the coating also has a single-phase structure, but with δ-MoN. The increase in the pressure of the reaction gas in the spray chamber is accompanied by an increase in the microhardness of the coating from 31 GPa (at 0.4 Pa) to 51 GPa (at 1.9 Pa). With increasing voltage on the substrate or substrate temperature, the microhardness of the coatings decreases.

In general, the nature of the change in the composition of the coating in the Mo-N system from the nitrogen pressure in the chamber has many features in common with the behavior of the Ti-system.

The microhardness of coatings corresponding to the α-Mo+γ-Mo_2N composition increases from ~13 GPa to 30–34 GPa with increasing γ-Mo_2N phase content, which also coincides quite closely with the data for coatings of the Ti-composition. But there is one significant difference, which, in this consideration, it is advisable to pay attention, namely — much lower thermal stability of molybdenum nitrides compared to titanium nitrides.

This, on the one hand, limits the allowable substrate temperatures and displacement potentials for the formation of coatings with sufficient microhardness [35], and on the other hand, is one of the reasons that makes it attractive for use in friction pairs. The latter is due to the fact that at high contact loads in friction pairs on micro-irregularities that accept the greatest local loads, and therefore primarily wear out, local temperatures can develop that are sufficient to cause the decay of molybdenum-nitrogen compounds.

In this case, it will be accompanied by a decrease in the hardness of the coating in these local areas, which, in turn, will reduce the wear of the coating, improve the serviceability of friction surfaces, the formation of such a surface structure that will adapt to certain conditions.

High tribological properties of coatings based on Mo-N are noted by the authors of many works [25, 36, 37].

As in the case of titanium nitride coatings, studies of the deposition of nitride vacuum arc coatings based on molybdenum were performed to determine the optimal parameters of the process of obtaining high quality Mo coatings on hardened steel with low tempering temperature, polished to V12 purity class.

These data are necessary to obtain further composite nano- and microlayer coatings based on molybdenum.

The coatings were obtained at an ionic current density $I = 10$ mA/cm^2, an accelerating substrate potential $U_n = -25$ V and a substrate temperature $T_n < 200$ °C. Nitrogen pressure during condensation $2.5 \cdot 10^{-3}$ mm Hg

Table 3.3 shows the results of experiments to determine the growth rate of coatings obtained on both stationary substrates and substrates that carry out planetary motion.

Table 3.3 The growth rate of MoN coatings of different composition

No.	Coating	The growth rate , V, µm/h	Notes
1	MoN	0.7	Fixed position
2	MoN	0.14	Planetary movement
3	Mo	0.2	Planetary movement

Based on the data on the growth rate of the coatings, data were entered into the control program of the Avinit plant to obtain Mo-MoN nanolayer coatings with a repeatability period of 20 nm and an equal thickness of the individual nanolayers.

The protocols of the automated control system of these processes are presented in Fig. 3.4.

The composition and some characteristics of the studied coatings are given in Table 3.4.

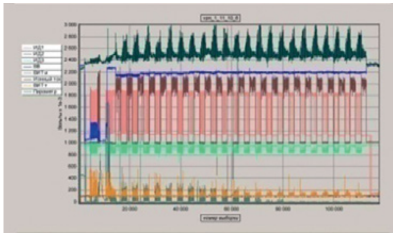


Fig. 3.4 Protocols of the automated control system are the process of obtaining Mo-MoN coatings with a repeatability period of 20 nm and equal thickness of individual nanolayers

Table 3.4 Characteristics of the samples

No.	The composition	The hardness of the base, HRC	Technological parameters			Properties of coatings		
			Programmed composition	T, °C	Nitrogen pressure, P, Pa	Coating thickness, µm	Microhardness coating, HV, MPa	Roughness, R_a, µm
Coating based on Mo-N								
1	Avinit C/P 200 (MoN)	59–60	Monolayers without separator	250	$1.5 \cdot 10^{-1}$	10.0	20000–22000	0.60 (8a)
2	Avinit C/P 200 (MoN)	59–60	Monolayer with separator	250	$1.5 \cdot 10^{-1}$	1.0	20000–23000	0.040 (11c)
3	Avinit C/P 210 (Mo-MoN)	59–60	Nanolayers with a repeatability period of 10 nm and a thickness of individual nanolayers of 2 nm and 8 nm	250	$1.5 \cdot 10^{-1}$	1.0	17000–19000	0.036 (12a)
4	Avinit C/P 220 (Mo-MoN)	59–60	Nanolayers with a repeatability period of 20 nm and an equal thickness of individual nanolayers	250	$1.5 \cdot 10^{-1}$	1.0	18000–20000	0.036 (12a)

When depositing nitride-molybdenum coatings without the use of separating devices, the microhardness of the coating with a thickness of

12 μm is 3150 kg/mm^2 and with increasing substrate temperature decreases to 2000 kg/mm^2.The surface roughness of the coatings corresponds to V 7–8 class when deposited on the polished to V 12 purity class surface of steel DIN 1.2379.

The experiments were performed on DIN 1.2379 steel samples with pure molybdenum coatings applied at different U_n values (25...200 V) at $T_n=(90...150$ °C).

Measurements of nanohardness using a nanohardness tester on Avinit C/P 210-m1 coatings with a thickness of 1.0 μm gave the following results – $HV=1500$–1800 Vickers, $H=1800$–2300 MPa, $E=200$–260 GPa, Poisson's ratio $K=0.30$.

The condensation process at temperatures not exceeding 150–200 °C does not significantly reduce the hardness of the original surface. Condensation at temperatures above 300 °C almost completely removes the effect of strengthening steel DIN 1.2379.

Regularities of changes in surface morphology (roughness) due to ion etching and condensate application show the need to reduce the time and temperature of ion bombardment and condensation in order to reduce the effects of surface etching and maintain strength characteristics in both ionic purification and condensation (Fig. 3.5).

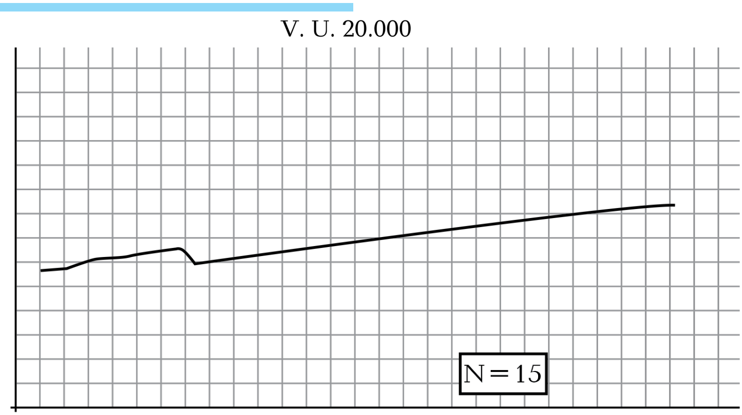


Fig. 3.5 Profilogram of the nanocomposite Ti-TiN coating

There is a tendency to increase the rate of condensation with increasing substrate temperature. The condensate is in a more equilibrium structural state due to the relaxation of the distortions of the crystal lattice, more equilibrium conditions for the formation of nitrides.

With increasing nitrogen pressure during condensation ($P_n>5{\cdot}10^{-3}$ mm Hg) there are almost no structural changes and microhardness of the coatings obtained at different values of the parameters U_n (15...70 V) and T_n (150...500 °C).

Increasing the energy of ions (U_n) during condensation is similar to the action of temperature and contributes to structurally more homogeneous and equilibrium materials.

3.2.3 Avinit C 700 coating (based on chromium nitrides)

Chromium nitrides, as well as molybdenum nitrides, have high values of microhardness and are widely used as reinforcing wear-resistant coatings both on the cutting tool and in the friction units of machine parts [30].

Studies on the possibility of applying nitride coatings and the dependence of their properties on the parameters of the condensation process [22, 38–40]. The parameters of the experiments were: nitrogen pressure (P_n), accelerating potential of the substrate (U_n), condensation temperature (T_n) at the values of the combustion current of the arc $I_g = 120$ A.

The coating material has an imperfect crystalline structure, as evidenced by very blurred diffraction maxima. In accordance with the change of phase composition and structural state, the values of microhardness of coatings change.

As the temperature and pressure of nitrogen increase, mainly Cr_2N_x and Cr are formed relaxation of crystal lattice distortions.

The phase composition and structural state of the condensate are largely determined by the energy of the ions. Experimental data, in particular, indicate that the condensates obtained at $T_n = 150$ °C, $U_n = -70$ V, $P_n = 10$ Pa have an extremely curved structure, the diffractograms recorded 2–3 maxima (say Cr, CrN, Cr_2N).

As the temperature rises to 500 °C, unidentified phases are recorded along with Cr, Cr_2N (CrN). The microhardness of such coatings decreases with increasing values of the substrate potential (ion energy) and temperature. Increasing the energy of ions (U_n) during condensation contributes to structurally more homogeneous coatings.

The patterns in the change of surface morphology depending on the condensation parameters are similar to those inherent in Mo-based coatings.

3.2.4 Avinit C 300 coating (based on Ti-Al-N)

Even a relatively simple Ti-system when considered from the point of view of multicomponent systems can have different heterostructures and, accordingly, different properties and different areas of application.

Naturally, an even greater variety of properties and expansion of applications can be expected from more complex systems.

In [41–47], numerous experimental studies of multi-component multilayer ion-plasma coatings based on the Ti-Al-N system were performed.

Ti-Al-N system coatings have high values of hardness, temperature resistance, etc. Such coatings as TiCrN, TiMoN, NbZrN are characterized by high thermodynamic stability, rather high values of hardness and viscosity, significantly exceeding their corresponding brittle oxides, carbides, and borides.

The coating (Ti-Al-N) is an example of a multicomponent coating, which in terms of prevalence has overtaken its predecessor TiN due to higher heat resistance, hardness, better tribotechnical characteristics.

Sequential replacement of titanium atoms by aluminum atoms in the titanium nitride lattice leads to the formation of the compound $(Ti_{1-x}Al_x)N$. Since the size of the aluminum atoms is smaller than the size of the titanium atoms, the lattice parameter of titanium nitride gradually decreases from 0.423 to 0.417.

The hardness of the coatings (Ti-Al-N) depends largely on the aluminum content. With an increase in aluminum content, the coating hardness increases from the values characteristic of TiN coatings to values close to 40 GPa at an aluminum concentration of 40–50 at % relative to titanium. With the further growth in the aluminum content in coatings up to 60 at. %, their hardness drops to ~33 GPa, then to ~21 GPa at a concentration of Al 70 at. % and with a further decrease in the concentration of aluminum approaches, accordingly, to the hardness of aluminum nitride coatings [39].

Similarly, the change in aluminum concentration is followed by the Young's modulus, which has a maximum value of about 650 GPa at an aluminum concentration of 50 at. %.

Similar are the dependencies of microhardness on the coating composition, which are given by authors of other works [39], although the absolute values of microhardness may differ from the above-given.

(Ti-Al-N) coatings have the advantages due to their being durable compared with many others.

This allows their operation at temperatures of 800–900 °C.

The upper temperature limit of these coatings operation depends on the content of Al, since it is the aluminum-oxygen compounds formed at high temperatures in the surface layers of the coating that ensure its heat resistance.

At the same time, the presence of aluminum in the coating has a positive effect on the magnitude of the friction coefficient, which makes the coating based on Ti-Al-N elements even more attractive for use as a high-potential material for friction couples [41–47].

Multilayer coatings are a series of layers of varying composition and alternating thickness.

The thickness of each layer can range from a few nanometers to several microns.

In [47], it is noted that the hardness and strength of the layer coating increases with the thickness of the individual layers.

Thus, if single-layer TiAlNCrN coatings have a hardness of 24 GPa, then multi-layer TiAlN-CrN coatings with a layer thickness of 15 and 6 nm, respectively, have a hardness of 35 GPa.

Layered composite coatings have advantages in a number of cases over monolayer coatings, for example, at friction units.

Composite layer coatings can combine an increased hardness with low wear rate of the counterbody.

This field of durable vacuum-arc coatings application is relatively less explored compared to its use as hardening ones, although this line of application is no less relevant in contemporary engineering and instrument engineering.

Table 3.5 provides the results of experiments to determine the coatings growth rates obtained on substrates performing planetary motion.

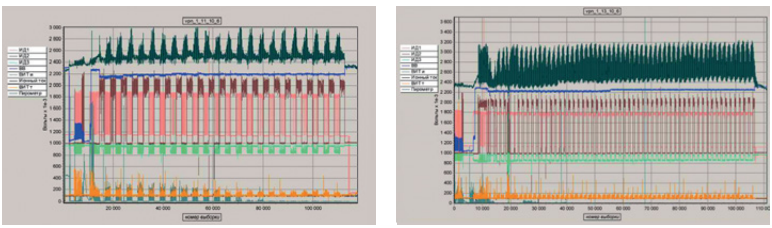
Table 3.5 The growth rate of different composition coatings

No.	Coatings	The growth rate, V, μm/h	Notes
1	Avinit C/P 300	0.7	Planetary motion
2	Avinit C/P 310	0.25	Planetary motion
3	Avinit C/P 320	0.25	Planetary motion
4	Avinit C/P 350	0.30	Planetary motion

Based on the data on the coatings growth rate, data was entered into the Avinit installation control program to obtain TiN-AlN nanolayer coatings.

The layers structure is provided by the time of successive stay of the surface coated in the area of action of each cathode and is determined by the distance L from the cathode to the surface, the ion current density of the arc sources, and the rotational speed of the rotary device.

The protocols of an automated control system for these processes are presented in Fig. 3.6.



a *b*

Fig. 3.6 A fragment of the protocol of the automated control system of the technological process of nanocoating TiN-AlN: *a* – TiN-AlN (50/50) nanocoating with a recurrence period of 20 nm and the similar thickness of individual nanolayers; *b* – TiN-AlN nanocoating (30/70) with a recurrence period of 12 nm and a thickness of individual nanolayers of 4 and 8 nm

Composition and some characteristics of the coatings under study are provided in Table 3.6.

Table 3.6 Characteristics of the samples

No.	The composition	The hardness of the base, HRC	Technological parameters			Properties of coatings		
			Programmable composition	T, °C	Nitrogen pressure, P, Pa	Thickness, µm	Microhardness, HV, MPa	Roughness, R_a, µm
1	2	3	4	5	6	7	8	9
The coatings based on Ti-Al-N (TiN-AlN)								
1	Avinit C/P 300	59–60	Multilayered	200	$3 \cdot 10^{-1}$	10.0	26000–30000	0.040 (11b)
2	Avinit C/P 310	59–60	Nanolayered with a repeatability period of 12 nm and a thickness of individual nanolayers of 4 and 8 nm	200	$3 \cdot 10^{-1}$	1.5	‡ 3000–3200 HV_{100} † 2500–3000 HV	0.036 (12a)
3	Avinit C/P 320	59–60	Nanolayered with a repeatability period of 12 nm and a thickness of individual nanolayers of 8 nm and 4 nm	200	$3 \cdot 10^{-1}$	1.5	‡ 3000–3500 HV_{100} † 3000–3500 HV	0.036 (12a)
4	Avinit C/P 350	59–60	Nanolayered with a repeatability period of 20 nm and an equal thickness of the individual nanolayers	200	$3 \cdot 10^{-1}$	1.5	26000–35000	0.036 (12a)
5	Avinit C/P 380	59–60	Nanolayered with a repeatability period of 20 nm and an equal thickness of the individual nanolayers	200	$3 \cdot 10^{-1}$	3–5	25000–35000	0.026 (12b)

Continuation of Table 3.6

1	2	3	4	5	6	7	8	9
6	Avinit C/P 410	59–60	Nanolayered with a repeatability period of 20 nm and an equal thickness of the individual nanolayers	200	$3 \cdot 10^{-1}$	3–5	15700–25500	0.025 (12b)
7	Avinit C/P 710	59–60	Nanolayered with a repeatability period of 20 nm and an equal thickness of the individual nanolayers	200	$3 \cdot 10^{-1}$	3–5	25000–30000	0.040 (11c)

‡ Measuring microhardness HV_{100} on witness-samples using microhardness tester.
† Measuring nanohardness using nanohardness tester.

To obtain the rigidly assembled coatings on precision surfaces it is very important to choose the proper mode and method of heating the openwork and to choose the right design of the intermediate layers.

Even slight deviations from the optimum technology can cause distortion of the parts to be covered.

Since the coating deposition operation is a finishing step, it often leads to the impossibility of restoring and completely rejecting complex unique structures.

Coatings are deposited at temperatures not exceeding 200 °C, which ensures preservation of the substrate mechanical properties and does not lead to a decrease in the hardness of the base — steel DIN 1.2379 (Table 3.6). In this case, the coatings have good adhesion to the base. Due to the use of special separating devices, the technology ensures the obtaining of vacuum-arc coatings with a sharply reduced fraction of the "drip" component, which allows virtually not to change the roughness of the original coated surface (Table 3.6).

Comparison of the substrate and the coating roughness indicates that after coating deposition to the samples with roughness corresponding to 12–13 purity grade, the surface roughness is practically unchanged, or there is a slight increase in roughness, which virtually does not go beyond one class according to the surface roughness grades classification.

The crystalline structure corresponded to the TiN structure with a lattice parameter close to the values of this compound.

According to X-ray studies, the size of coherent scattering (OCD) regions in the coating was equal to 32 nm. This value matches well with the sizes of the individual TiN and AlN nanolayers calculated based on the nanolayer growth rate per one revolution, which was ~35 nm, which, in general, confirms the presence of the nanolayer structure in accordance with the coating formation flow chart.

Fig. 3.7 shows the results of Avinit C 320 coating electron-probe X-ray microanalysis (EPRMA) performed for three elements: aluminum, iron and titanium on the Avinit C 320 sample when scanning the sample with an electron beam (diameter of the electronic probe ∅ = 30 nm, characteristic radiation is recorded from the surface layer of the sample at a depth of 1 μm).

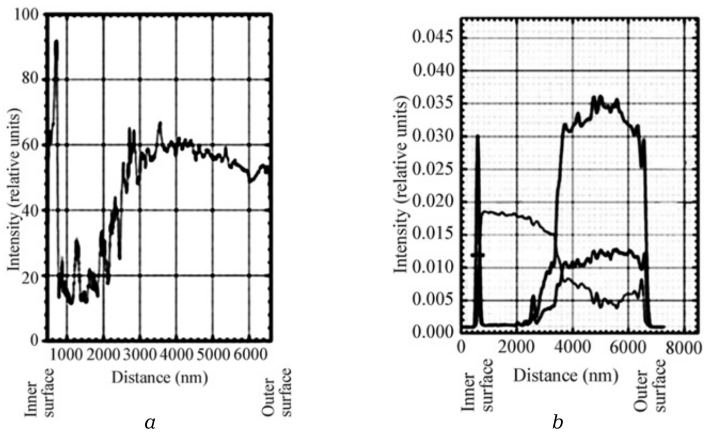


Fig. 3.7 Distribution of characteristic X-ray radiation of element atoms in Avinit C 320 coating: *a* – at. % Ti; *b* – at. % Al

When scanning from the outer surface to the inner surface, curves begin with peaks of the intensities of titanium and aluminum caused by the characteristic radiation of these metals deposited on the outer cylindrical part of the sample.

The peaks of characteristic radiation of Al and Ti with intensities of the same order are observed at the interface of the inner butt and cylindrical surfaces.

Al and Ti distributions throughout the analyzed surface are qualitatively close to each other.

The results of metal-physical measurements of the coating Avinit C/P 310-n1 on a scanning electron microscope JSM T-300 are shown in Fig. 3.8, 3.9.

Microhardness values of the coated specimens surfaces provided in Table 3.6, indicate the cumulative effect of increasing the microhardness of the surface due to the harder coating, rather than the actual values of the coatings microhardness, since nanohardness testers are required to determine the microhardness of thin coatings (<4 μm).

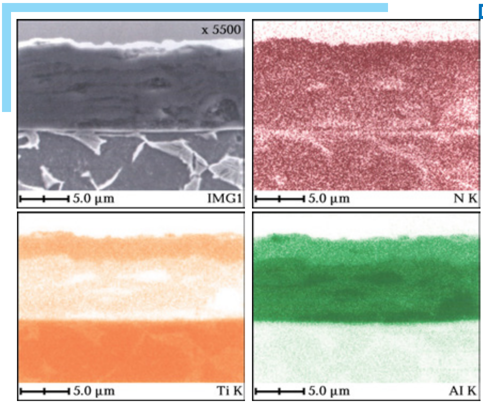


Fig. 3.8 Appearance of a covering of Avinit C/P 310 (cross section) in the mode of mapping of a site of a coating

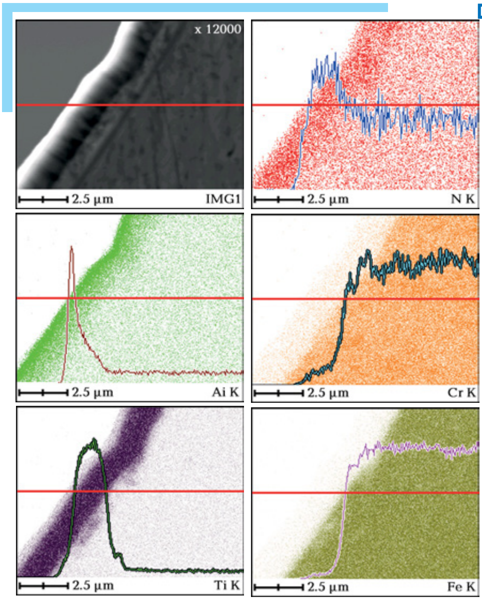


Fig. 3.9 Appearance of Avinit C/P 310-n1 coating in line analysis mode

If for Ti-N system coatings, the microhardness value has a more constant value for different modes, and on average has value close to 2300–2400 MPa, then for the Mo-N and Ti-Al-N system coatings its value can have significant fluctuations as shown above, and it would be desirable to have more detailed information on this. To determine the thin coatings microhardness (<4 μm), nanohardness measurements were performed using a CSM (Switzerland) nanohardness tester (loading rate of 20.00 mH/min, max depth of 100.00 nm at a load of 0.6 G), processing of results using standard software based on the application of the Oliver-Farah model.

The measurements of microhardness and Young's modulus in Avinit C 320 coatings with a thickness of 1.4 μm gave values of $H_v = 1.600–2300$ kg/mm^2, $E = 250–300$ GPa, Poisson's coefficient $K = 0.30$ (load diagrams are provided in Fig. 3.10).

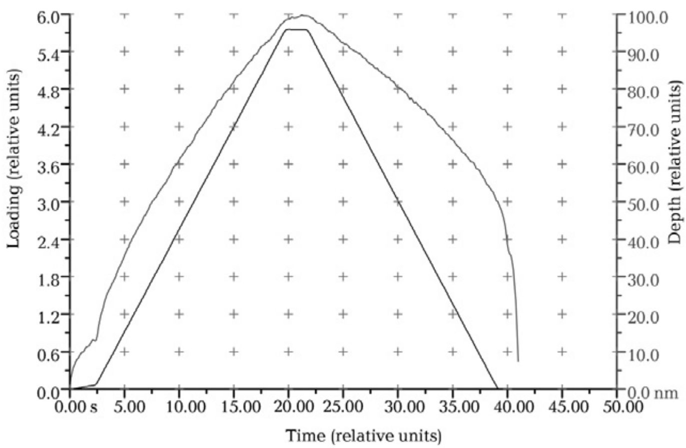


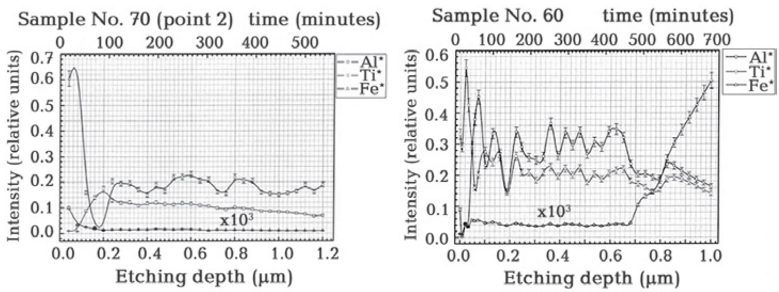
Fig. 3.10 Load diagram for measuring nanohardness and Young's modulus of Avinit C 320 coating

It should be noted that in the Oliver-Fahr model, the Young's modulus of the coating and the bases are assumed to be the identical and therefore, the calculated values may be slightly underestimated.

Performed nanohardness measurements indicate that even in thin layers of hard and superhard coatings, where the use of conventional methods of microhardness measuring using microhardness tester PMT-3 is impossible (the coating thickness in order to obtain reliable information must be at least 5 μm), the same high hardness values are obtained, as in the thick layers. This allows to confidently assert that many of the technological improvements we have made for thick coatings, can be successfully transferred to thin coatings for precision surfaces.

Metallographic studies of Avinit-type coatings were performed using the methods of secondary ion mass spectrometry (SIMS), electron probe X-ray microanalysis (EPMA), scanning electron microscopy (SEM).

Fig. 3.11 shows the dependences of the secondary Al^+, Ti^+ ions currents on the sputtering time for the Avinit C 320 coating, and, respectively, and on the depth of the component distribution profile.



a *b*

Fig. 3.11 Dependences of secondary Al^+, Ti^+ ions currents on sputtering time: *a* — Avinit C 320 coating; *b* — Avinit C 310 coating

The change in the current of the secondary ions in the both experiments characterizes the change in the concentration of the corresponding elements in the sample's depth by sputtering the near-surface region with a beam of primary Ar^+ ions.

From the obtained dependences it follows, that the top layer of the coating has an increased concentration of aluminum, which decreases with depth.

Similar dependences with respect of aluminum and titanium distribution profiles in the near-surface area of the samples with the deposited functional coating of Avinit C 310 are shown in Fig. 3.11b. Synchronous changes in the intensities of currents Al^+ and Ti^+ from a depth of ~1.8 μm are associated with the technology of coating formation.

Thus, the experimental results confirm the possibility of low-temperature deposition of durable Avinit C very hard coatings based on metal nitrides in modes that provide good adhesion to substrate materials (DIN 1.2379 steel with a precision surface $R_a = 0.025$ μm) without a significant reduction in the steel strength characteristics (<200 °C) and without compromising the purity grade of the original surface.

The studies carried out made it possible to choose the temperature-time parameters for obtaining Avinit C hardening coatings to increase the wear resistance of the working surfaces of precision friction couples, providing a coating of a given composition with a thickness of 1–3 microns, and to develop software products for obtaining such coatings on the Avinit equipment and elaborating technologies for deposition of multicomponent multi-layer coatings on real parts of the serially produced units.

3.3 Research of friction and wear characteristics of coatings

Friction

Numerous tribological studies show that vacuum-plasma coatings have high wear resistance and tribological characteristics. We conducted in-depth tribological tests of advanced functional multi-component multilayer coatings Avinit [17, 48–52]. The parameters of Avinit coatings during tribological tests for abrasion resistance and wear are given in Table 3.7.

Table 3.7 The parameters of Avinit coatings

1	Microlayer coatings Avinit C 320	HV = 3500 kgf/mm^2, h = 1–2 µm
2	Multilayer coatings Avinit C 210	HV = 2300 kgf/mm^2, h = 1–2 µm
3	Multilayer coatings Avinit C 220	HV = 2300 kgf/mm^2, h = 20 µm
4	Multilayer coatings Avinit C 220	HV = 2300 kgf/mm^2, h = 16 µm
5	Nanolayer coatings Avinit C 320	HV = 3500 kgf/mm^2, h = 1–2 µm
6	Multilayer coatings Avinit C 350	HV = 3500 kgf/mm^2, h = 20 µm

The values of the coefficients of friction in tribopairs, recorded during tests for abrasion resistance and wear, are shown in Fig. 3.12.

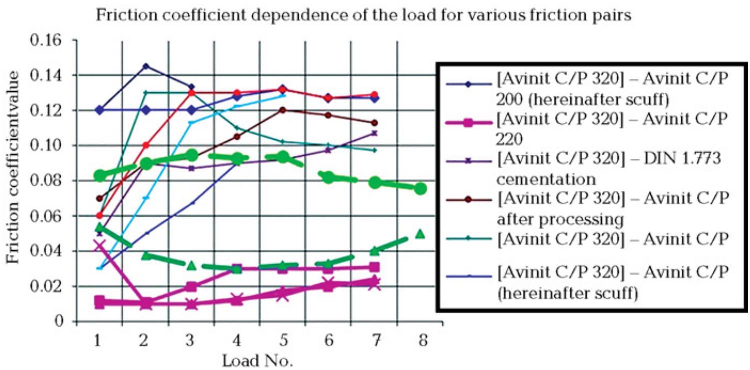


Fig. 3.12 Friction factors

After testing all friction pairs, there are no signs of increased wear or adhesion of the working surfaces of the samples with Avinit coatings. According to the tribological studies of improved coatings such as Avinit, coating deposition effectively helps to increase the pair resistance to scoring while increasing the scoring development P_{cr} value. The use of multilayer coatings (e.g., Avinit C 110 of TiN-Ti type) leads to an increase in P_{cr} compared to monolayer coatings (eg, Avinit C 100 of TiN-Ti type).

Particularly, effective coatings are those based on Avinit C 220 (MoN-Mo type) which have the highest P_{cr} values. All improved coatings had low friction coefficients at loads up to 2.0 kN. This is evidenced not only by increasing the load during the tests to the limit, but also by the course of the dependence of the friction coefficients on the load, which after some increase in load to 0.6–0.8 kN decreased to a maximum load of 2 kN. The values of the friction coefficients for all types of coatings are close enough, and at loads greater than 1.0 kN they are within the range from 0.06 to 0.1. The lowest friction coefficient is the small pair — Avinit C 320 coating-Avinit C 220 coating. The value of the friction coefficient of the pair did not exceed 0.095 within the whole load range, and at the maximum load it equaled 0.065, which corresponds to the minimum value obtained for the friction tribocouples with the coatings under study. All coatings in the tests showed high resistance to wear, the value of which did not exceed 0.8 µm. The entire obtained array of tribological data for the studied coatings in averaged form is presented in Fig. 3.13.

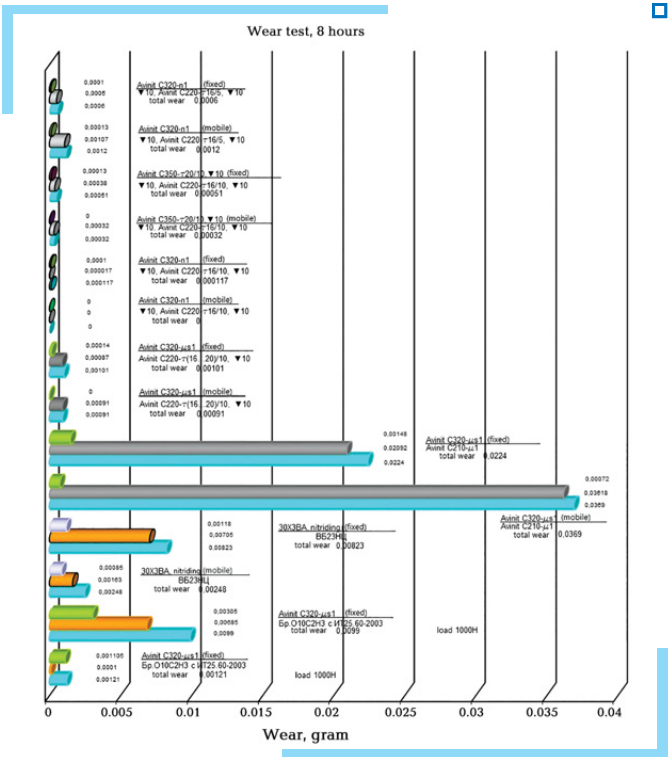


Fig. 3.13 Generalized tribological test results

Fatigue

Intensively wearing elements of TPP equipment, generally, are exposed to significant dynamic loads. For securing effective work of the equipment's elements that are exposed to shock, corrosive and erosive loads a matter of importance is the correct selection of technologies of the working surfaces' chemical-thermal treatment, composition of hardening coatings, and these coatings deposition technologies. There have been conducted fatigue strength tests of the 1.4021 Ge steel samples without coatings and with wear-resistant multilayer coatings [13, 53, 54].

The results have shown:

- coating significantly enhance fatigue strength of the DIN 1.2379 steel samples – lifetime of the samples with coatings is several times longer comparing to those without coatings;
- endurance limit in the air of the coated samples is approximately by 15–25 % higher than endurance limit of the initial samples.

In [13, 54] there have been conducted studies of the developed ion-implantation and vacuum-plasma processes of surfaces modification with various morphology and structural-phase composition to develop technologies of the surfaces creation in order to significantly enhance performance characteristics of the heavily loaded details.

Conducted fatigue tests [13] of DIN 1.2379 steels resulted in:

1. The 1.4021 Ge steel samples endurance limit in the initial state is 320 MPa and endurance limit of the coated samples (ion modification + multilayer coating TiN) accounts for 350 MPa (endurance limit increasing by 9 %).

2. The 1.4021 Ge steel samples exposed to corrosive environment have endurance limit in the initial state is 180 MPa and endurance limit of the coated samples (ion modification + multilayer coating TiN) accounts for 250 MPa (endurance limit increasing by 38.9 %).

Developed technologies of ion-implantation with subsequent deposition of coatings based on titanium nitrides secure enhancing of:

- resistance to drop erosion 1.35–1.71 times;
- corrosion resistance 2–3 times;
- fretting resistance up to 4 times for titanium alloys DIN 3.7165;
- operating time without scuffing under fretting condition 1.3–2.5 times;
- surface hardness over 15 000 MPa;
- coating adhesion over 80 MPa.

Corrosion

Studying corrosion resistance of TiN monolayer coatings for working blades of steam turbines in gas abrasive and moisture-steam environment under temperatures up to 500–540 °C described, that their protective properties, as anticorrosion coatings, are low despite the coating thickness due to the porous structure of this kind of coatings. Multilayer vacuum-plasma coatings based on refractory nitrides of metals have much better anticorrosion properties even in very aggressive environments. The work [54] are devoted to the multilayer coatings wherein corrosion-resistant metal titanium was used as a primary layer (sublayer). Corrosion tests were conducted with the 1.4021 Ge steel samples in both coated (ion modification + multilayer coating TiN) and initial states according to expedited methodology. These studies have shown that multilayer coatings obtained according to technology of ion implantation with subsequent coating deposition based on titanium nitrides ensure 2–3 times corrosion resistance enhancing [54].

Erosion

Studying erosion resistance of the TiN-based coatings conducted within the temperature interval of 100 to 330 °C [12], allowed determining the optimal range of temperature values of coating formation. With the increasing temperature of coatings formation process erosion resistance increases. Minimal allowable values of relative erosion resistance, in terms of practical application, were obtained under the temperature of 200 °C. Increasing of the substrate temperature over 330 °C is impractical given that further erosion resistance increasing is low, however, high temperature level can cause unwanted changes of metal mechanical properties.

Studies to determine the impact of ion-plasma coatings' thickness on erosion resistance showed that increasing coating thickness leads to enhancing of its protective properties and, therefore, to enhancing erosion resistance of the turbine blades steel.

Besides the forming temperature and coatings' thickness, conditions of the coated surface have significant impact on its exploitation properties. Capability of preliminary treatment of the surface with high-energy ions is a distinctive feature of all ion-plasma technologies. Due to surface treatment with a flow of high-energy particles, it is possible to achieve modifying of materials surface properties that usually positively affect properties of the formed coatings. Duration of surface treatment with argon ions significantly affects erosion resistance of formed coatings of both small thickness (2.5 μm) and large thickness (9 μm and more) [12].

The results of metal-physical and tribological studies performed are the basis for the selection of coating materials and the development of new designs of anti-friction wear-resistant coatings to improve the efficiency of friction couples in the "coating-steel" and "coating-coating" systems, as well as to develop their deposition processes.

3.4 Plasma chemical heat treatment Avinit N

At creation of new materials with ultrahigh characteristics technologies of modification of surface layers of contacting materials and drawing functional coatings [55, 56] are more and more successfully used.

The service life of parts of various devices, units in many cases is determined by the ability of the material to withstand the fatigue failure of working surfaces, which is characterized by such a value as the contact strength of the material.

It is known that the contact strength, in the General case, can be increased by reducing the mechanical and thermal effects on the contact surfaces due to their deformation and friction. Traditionally, this problem is solved by increasing the hardness of the contact surfaces, the use of antifriction coatings and lubricants.

The most common is the chemical-thermal method of cementing the surface of materials.

However, in the case of cementation, hardening occurs with the simultaneous accumulation of energy at the interface of the grains, which subsequently lead to the growth of microcracks. In addition, the release of energy during the contact interaction of the surfaces leads to their heating and destruction in the cemented layers of carbides, resulting in a decrease in contact strength.

Nitriding methods as a method of strengthening the parts of machines and tools are also used to increase the contact strength of materials, [57–61] and are one of the effective and common technological methods to ensure the functional properties of many parts. Nitriding is an important element of turbine blade manufacturing technology.

Standard technologies for the production of many industrial parts designed to work in high-precision gears, hinges and plain bearings, which are made of steels such as DIN 1.773, CrMoV5-5, etc., include pre-nitriding by traditional methods, further grinding and polishing of work surfaces after nitriding, and finally, the application of wear-resistant coatings.

There are a number of traditional methods of nitriding, long and widely used in industry — gas nitriding, liquid nitriding, ionic nitriding in the glow discharge plasma, etc. [57–61].

Typically, the nitriding process involves heating the parts to a given temperature in the range of 500...600 °C and subsequent exposure in a saturating atmosphere for diffusion saturation of the surface of the parts with nitrogen. At the same time it is possible to increase plasticity and resistance of a bully of a superficial nitride zone of the processed details.

Among the advantages should be noted:

- high hardness (up to 1300 HV), which is achieved without hardening;
- slight deformation of parts in comparison with other methods of hardening;
- heat resistance of the surface layer up to 500–600 °C;
- high wear resistance;
- corrosion resistance (especially in the atmosphere);
- high fatigue endurance of nitrided details.

The disadvantages of traditional methods include:

- long duration of the saturation process (up to 100 hours);
- the need to use special expensive steels, which are also non-technological at different stages of manufacturing parts;
- low in comparison with cemented details contact durability;
- very high environmental friendliness of industrial methods;
- fragility of the surface layer and reduced viscosity of nitrided parts;
- instability of nitriding results in its implementation in industry.

The main disadvantages of these technologies are the presence on the nitrided surface of secondary brittle iron nitrides Fe_4N both on the surface and in the grains of the reinforced layer. This contributes to the internal stress, which leads to warping of the coating with changes in the geometric dimensions of the parts.

Diffusion saturation methods based on the use of plasma processes are also widespread [62–65].

In the known methods of ionic nitriding in the glow discharge plasma, diffusion saturation is carried out in a reactive nitrogen-gas medium, or in a glow discharge using a gas-discharge plasma generator with an incandescent cathode [62].

It is possible to use vacuum ion-plasma treatment of parts [63] in gas-discharge plasma containing argon and hydrogen ions, diffusion saturation, and product processing and diffusion saturation are carried out simultaneously in gas-discharge plasma, which additionally contains ions of carbon or carbon and nitrogen.

This allows to form in the surface layer of dispersed carbides or carbonitrides and, thus, increase its hardness. Additional bombardment with inert gas (argon) ions contained in the gas-discharge plasma increases the diffusion rate, and hence increases the thickness of the reinforced surface layer.

The disadvantages of ion-plasma methods are the low productivity of the nitriding and carbonitriding process, the formation on the surface of a solid layer of iron nitrides, the narrow scope of their application, as it is limited only by nitrided alloys (mainly structural steels with low alloying), subsequently on nitrided surfaces having a chemical affinity with the carbon or carbonitized layer of the substrate, and in this case the diffusion saturation may even lead to a decrease in the bond strength of the coatings with the substrate.

Nevertheless, nitriding processes to increase contact endurance have significant prospects in comparison with traditionally used cementation processes in terms of increasing surface hardness, especially by eliminating machining after nitriding.

Given the undeniable possible benefits of nitriding in the creation of new structural materials, the search for improved methods of nitriding remains relevant.

3.4.1. Plasma precision nitriding Avinit N

In [55, 64, 65] we developed a process of strengthening and increasing the wear resistance of steel and alloy products by plasma precision nitriding Avinit. Plasma precision nitriding Avinit N is a process of chemical-thermal treatment of machine parts, diffusion saturation of the surface layer with nitrogen in high-density ($n \geq 10^{10}$–10^{11} cm^{-3}) low-temperature strongly non-equilibrium plasma of nitrogen and argon.

The source of high-density plasma is a gas plasma generator of the Avinit installation [15]. The cathode of the discharge in such a plasma generator is the cathode of a double arc discharge, isolated from the chamber by a special separator that does not allow the flow of metal, but allows to create a high-density plasma in the chamber. The discharge current is closed at the anode with magnetic focusing.

The essence of plasma nitriding

The parts placed in the vacuum chamber are treated with high-density plasma excited by a plasma generator in a discharged inert gas medium. After heating to operating temperature, the plasma-forming gas in the chamber is replaced by nitrogen, which leads to the formation on the surface of the parts of the nitrided layer.

By changing the pressure, temperature, discharge parameters and holding time, it is possible to obtain layers of a given structure and phase composition, providing the necessary properties of nitrided surfaces.

The method of Avinit N is carried out as follows.

The method includes pre-ion-plasma treatment of steel parts and alloys in the plasma of a glow discharge of inert gas — argon at a pressure of $3 \cdot 10^{-4}$–$7 \cdot 10^{-4}$ mm Hg and a current density of 3.5 mA/cm^2 for 30 min, gradually increasing the negative bias voltage on the part to 1000–1200 V.

In the chamber of the installation, which contains a source of gas-discharge plasma (gas plasma generator), create a preliminary vacuum of $5 \cdot 10^{-5}$ mm Hg. Argon is introduced into the chamber through a gas plasma generator to a pressure of $1 \cdot 10^{-3}$–$2 \cdot 10^{-3}$ mm Hg and changing the incandescent current of the cathode from 0 to 200 A regulate the discharge current of the plasma generator and the density of the gas-discharge plasma.

The parts give rotation, give it a bias potential, gradually changing its value from 0 to 500...600 V, and carry out ionic surface cleaning. The intensity of ionic purification and the heating temperature of the part are regulated by changing the discharge current of the gas plasma generator, ie the current density of argon ions is from 0 to 5 mA/cm^2.

Exposure to plasma leads to heating of parts. The products are heated to a given temperature in the range of 400...500 °C in a gas-discharge plasma of argon formed by a gas plasma generator.

Then the plasma-forming gas argon is replaced by a gas mixture of Ar + 50 % N_2, supplied to the gas plasma generator at a pressure of

$1.5 \cdot 10^{-3}$ mm Hg. By increasing the bias potential to 600 V increase the temperature of the samples and conduct further isothermal exposure for 2 hours in the range of 400...500 °C in a gas-discharge plasma of argon and nitrogen.

Forming a gas plasma generator, a plasma stream containing nitrogen ions, at temperatures of 500...600 °C, carries out its own nitriding – diffusion saturation of the surface in high-density gas-discharge nitrogen plasma.

By changing the pressure, temperature and holding time, the parameters of the discharge, iti s possible to obtain layers of a given structure and phase composition, providing the specified properties of nitrided surfaces.

In the course of nitriding in the nitrided layer as a result of diffusion of nitrogen and the proceeding phase transformations there are the concentration fields causing increase in volume therefore there are the pressures promoting warping of details.

The porosity and fragility of the layer is manifested the earlier and to a greater extent, the earlier the nitrides are formed and the more intense is the further nitrogen saturation.

The initial stage of formation of the surface layer during nitriding is of great practical importance. It is at this stage that the proportion of the solid-solution mechanism should be increased and the nitride formation reaction, especially with very high nitrogen content, should be reduced to a minimum, and preferably completely prevented. This reduces the duration of nitriding, because the diffusion mobility of nitrogen in solid solution is much greater than its diffusion in other phases and eliminates or significantly reduces the possibility of porosity and fragility of the surface layer, as well as the formation of a uniformly reinforced layer, which is extremely important preserving the original geometric dimensions.

The method of nitriding is implemented in stages as follows.

Carry out heating of products to the set temperature in the range of 400...500 °C in gas-discharge plasma of argon formed by the gas plasma generator.

Carry out the following exposure in a gas-discharge plasma of argon and nitrogen, which is also formed by a gas plasma generator.

In the third stage, the diffusion saturation of the product surface is carried out at temperatures of 500...600 °C in a saturating nitrogen atmosphere, while the gas-discharge plasma contains nitrogen ions and is also formed by a gas plasma generator.

Experiments results

The experiments were carried out on samples of widely used steels 34NiCrMoV14-5, DIN 1.773, 4CrMoV5-5.

The samples were pre-ground and polished with a 1/0 diamond paste to grade 10 roughness.

To determine the characteristics of changing the geometric dimensions of the samples after nitriding, control samples were established made in the form of cylinders ∅ 20 mm of the same steel, which were subject to completely similar pre-heat treatment.

After diffusion saturation, the samples and control samples were examined to study the properties of the modified surface layer. A Raytek pyrometer was used to measure the temperature of the parts. Metallographic studies and determination of material parameters (layers thickness, uniformity, presence of defects, and structure of the material itself) were performed on MMP-4 and Tesa Visio 300 gL microscopes.

The microhardness of the layers was measured using a BUEHLER microhardness tester at a load of 50 g.

Measurements of the geometric dimensions of the control samples were performed with accuracy up to 0.5 μm before and after nitriding.

Plasma parameters (ion current, ion density, current-voltage characteristics, spectral characteristics) were continuously monitored and archived using a "PlasmaMeter" plasmometer and "PlasmaSpectr" spectrometer (Fig. 3.14, 3.15).

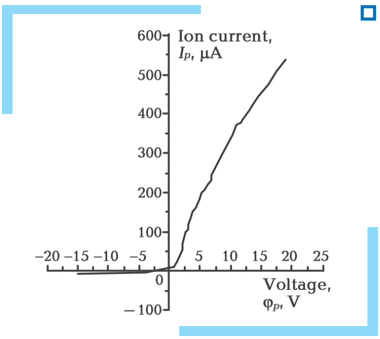


Fig. 3.14 Volt-ampere characteristic of a double arc discharge probe

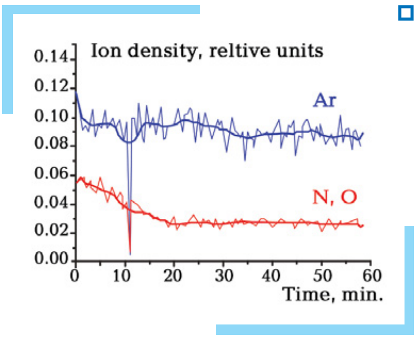


Fig. 3.15 Spectral parameters of plasma

Typical volt-ampere characteristic (VAC) of a double arc discharge probe at a nitrogen pressure of $1.2 \cdot 10^{-2}$ Torr, which was used to calculate the plasma parameters (ion current, plasma density, electron temperature, degree of ionization of the flux).

Nitriding of steel 34NiCrMoV14-5

Steel 34NiCrMoV14-5 samples and control samples from the same steel were used as substrates. After preliminary chemical purification in gasoline and distillation alcohol, the samples were placed in a vacuum chamber of the Avinit unit [15], in which a gas plasma generator was installed.

The samples were fixed in the center of the installation rotary table and they were rotated at an angular velocity of 2 rpm.

The chamber was pumped off to a pressure of $5{\cdot}10^{-5}$ mm Hg, then the argon was introduced to achieve a pressure of $3{\cdot}10^{-3}$–$7{\cdot}10^{-3}$ mm Hg, a glowing discharge of argon was ignited and ion-plasma treatment of the samples carried out for 30 min at a displacement potential of 1.000–1.200 V and a current density of 3–5 mA/cm^2.

The parts heating to a temperature of 400...500 °C was carried out in argon gas-discharge plasma produced by the gas plasma generator.

Argon was introduced into the vacuum chamber through a gas plasma generator to a pressure of $1{\cdot}10^{-3}$–$2{\cdot}10^{-3}$ mm Hg.

At the current of plasma generator cathode 100 A, the temperature of the samples was brought to 400...500 °C within 1 hour by gently regulating the negative displacement potential up to 400–500 V.

Plasma-producing argon gas was replaced in the chamber by a gas mixture of Ar + 50 % N2, which was supplied into the gas plasma generator at a pressure of $1.5{\cdot}10^{-3}$ mm Hg.

The following isothermal holding was carried out for 2 hours within the range of 400... 500 °C in argon and nitrogen gas-discharge plasma.

By increasing the displacement potential to 600 V, the samples temperature was brought to 530 °C.

A plasma stream containing only nitrogen ions was produced by the gas plasma generator.

Under these conditions, a direct nitriding was carried out in the fourth stage – diffusion saturation of the surface in high-density nitrogen gas-discharge plasma. Sample holding time – 2...3 hours.

The samples obtained were investigated in order to study the properties of the modified surface layer and to measure the geometric dimensions of the control samples.

The microstructure of the nitrided layer on 34NiCrMoV14-5 steel is shown in Fig. 3.16, 3.17.

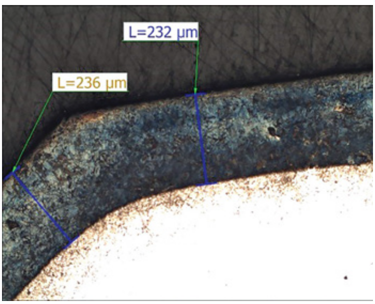


Fig. 3.16 Depth of nitrided layer on 34NiCrMoV14-5 steel (×500)

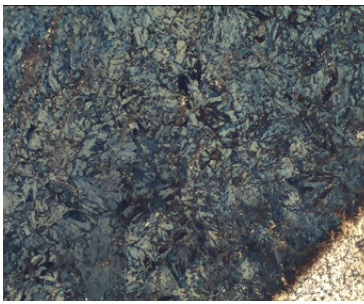


Fig. 3.17 The microstructure of the nitrided layer on 34NiCrMoV14-5 steel (×500)

The hardness value of the base metal after nitriding does not change $H_{\mu} = 350...370$.

The surface layer hardness of steel 34NiCrMoV14-5 after diffusion saturation with nitrogen increased to 830 HV.

In this case, the depth of the nitrided layer is 230...250 μm when nitriding at a temperature of 530 °C for 2 h, i.e., the nitriding efficiency is 4–6 times higher than with traditional nitriding methods.

Study of the nitrided layer microstructure (Fig. 3.17) indicates a uniform structure and the complete absence of a brittle surface layer characteristic of traditional nitriding methods.

Nitriding of steel DIN 1.8509 (Fig. 3.18)

Fig. 3.18 Appearance of the sample for plasma precision nitriding Avinit N steel DIN 1.8509

Metallographic studies of nitrided steel DIN 1.8509 are shown in Fig. 3.19. The average value of the hardness of the base metal after nitriding is: $H_{\mu} = 341.7$ (34.6 HRC). Microstructural analysis of the sections showed the presence of a nitrided layer on all surfaces. The thickness of the nitrided layer was — 0.41...0.43 mm.

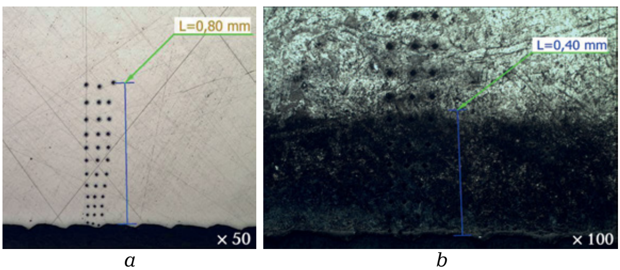


Fig. 3.19 Impressions of microhardness measurements: *a* – not etched section; *b* – etched section

The places of hardness measurements are shown in Fig. 3.19. The results are given in Table 3.8.

Table 3.8 The results of hardness measurements

№	Distance from the edge, mm	Hardness – H_μ, (in brackets – HRC)
1	0.01	630 (56.0)
2	0.08	672.3 (53.7)
3	0.15	513.6 (50.0)
4	0.22	445.5 (44.9)
5	0.29	424.5 (43.1)
6	0.36	420.4 (42.7)
7	0.43	401.1 (40.9)
8	0.50	350.5 (35.6)
9	0.57	344.5 (34.9)
10	0.64	338.6 (34.2)

Nitriding of steel DIN 1.773

Steel samples and control samples of the same steel were used as substrates (Fig. 3.20).

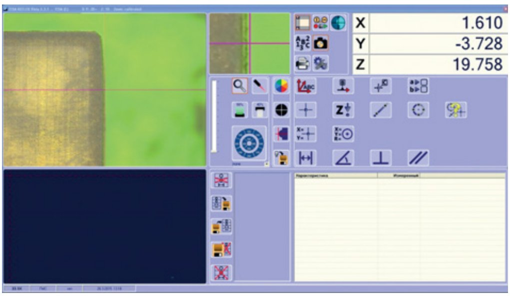


Fig. 3.20 Nitriding of steel DIN 1.773

The results of measurements of the hardness of the nitrided layer are given in Table. 3.9.

Table 3.9 The results of measurements of the hardness of the nitrided layer

№	Distance from the edge, mm	Hardness – H_μ
1	2	3
1	0.025	679
2	0.05	546

Continuation of Table 3.9

1	2	3
3	0.10	555.5
4	0.15	550.5
5	0.20	527.5
6	0.25	514.5
7	0.30	485.5

The depth of the nitrided layer was 230...250 μm when nitriding at a temperature of 530 °C for 2 h, i.e., the nitriding efficiency was 4–5 times higher than with traditional nitriding methods.

The hardness of the DIN 1.773 nitrided steel layer was 790 HV.

The hardness value of the base metal after nitriding does not change $H_{\mu} = 350...370$.

The nitrided layer has a uniform structure, without any fragile surface layer (Fig. 3.21, 3.22).

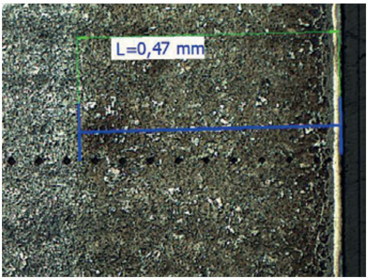


Fig. 3.21 Depth of nitrided layer

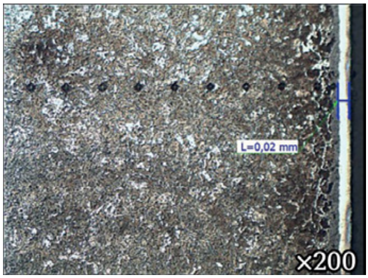


Fig. 3.22 Microstructure in the zone of the nitrided layer

Measurements of the control samples geometric dimensions indicate their invariability within the accuracy of 1–2 μm.

Nitriding of steel 24CrMoV5-5

The samples of 24CrMoV5-5 steel and control samples of the same steel were nitrided.

The modes and the time of nitriding were similar to those used in 1 method. The appearance of the nitrided layer on 24CrMoV5-5 steel is shown in Fig. 3.23.

Etched and unetched polished sections of the nitrided layer on 24CrMoV5-5 steel and hardness measuring points are shown in Fig. 3.24, 3.25.

The hardness of the base metal after nitriding does not change – $H_\mu = 350...370$.

The surface hardness of the 24CrMoV5-5 steel layer after diffusion saturation with nitrogen increased to 970 HV.

In this case, the depth of the nitrided layer was 260...280 μm when nitrided at a temperature of 530 °C for 2 h, i.e., the nitriding efficiency was 4–6 times higher than in traditional nitriding methods.

The study of the nitrided layer microstructure (Fig. 3.25) exhibits a uniform structure and the complete absence of a brittle surface layer, characteristic of traditional nitriding methods.

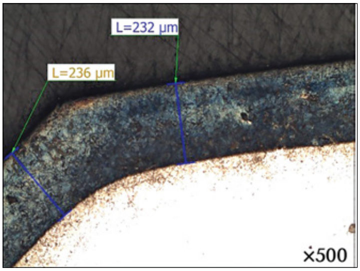


Fig. 3.23 The nitrided layer depth

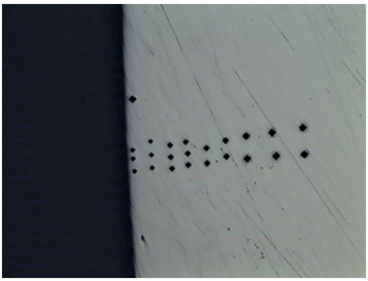


Fig. 3.24 Imprints of microhardness measurements (unetched polished section)

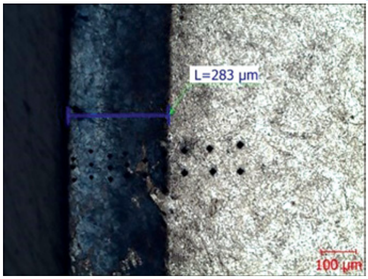


Fig. 3.25 Imprints of microhardness measurements (etched polished)

Studies of the effect of Avinit N plasma nitriding on the geometric dimensions of complex geometry parts have been performed.

Measurement of evolvement surfaces was performed on a Wenzel LH65 control and measuring machine using surface points plotted on a 3-D model of the part.

The obtained data indicate that Avinit N plasma nitriding does not change the original geometry of evolvement surfaces with an accuracy of 0.002 mm.

Avinit N plasma nitriding of parts from titanium and titanium alloys in glow discharge plasma

Avinit N plasma nitriding method was used to nitride ground and polished DIN 3.7165 titanium alloy samples and control samples from the same alloy.

The nitriding temperature was 900 °C, the nitriding time was 4 hours. On the sample of the titanium alloy DIN 3.7165 (Fig. 3.26) the structure and thickness of the nitrided layer, the nitrogen content and the change in the microhardness were studied. The microstructure of the sample outside the area of the nitrided layer is — α solid Ti solution (Fig. 3.27, 3.28).

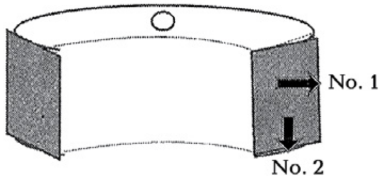


Fig. 3.26 Prototype of the titanium alloy DIN 3.7165

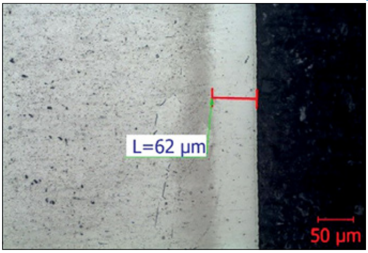


Fig. 3.27 The microstructure of the sample. Side No. 1 (×200)

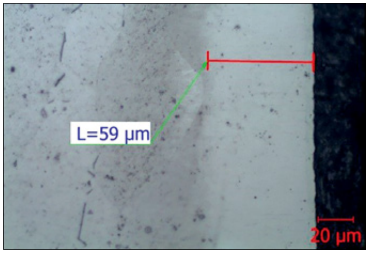


Fig. 3.28 The microstructure of the sample. Side No. 2 (×500)

The hardness of the nitrided layer was 950 HV and the depth of the nitrided layer was 50 μm.

The nitrided layer has a uniform structure, without any fragile surface layer. The hardness value of the base metal after nitriding does not change.

The magnitudes of change in the hardness of the nitrided layer, starting from the edge, are presented in Fig. 3.29, 3.30.

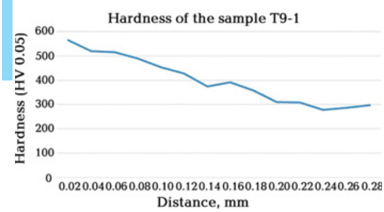


Fig. 3.29 The magnitudes of change in the hardness of the nitrided layer

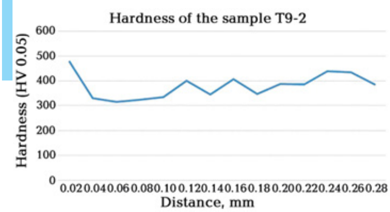


Fig. 3.30 The magnitudes of change in the hardness of the nitrided layer

Appearance of microhardness measurements imprints represented in Fig. 3.31–3.34.

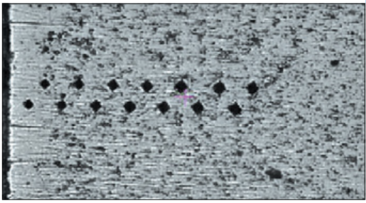


Fig. 3.31 Appearance of micro-hardness measurements imprints. Side No.1

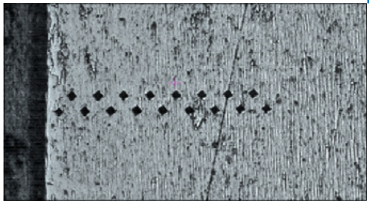


Fig. 3.32 Appearance of micro-hardness measurements imprints. Side No.2

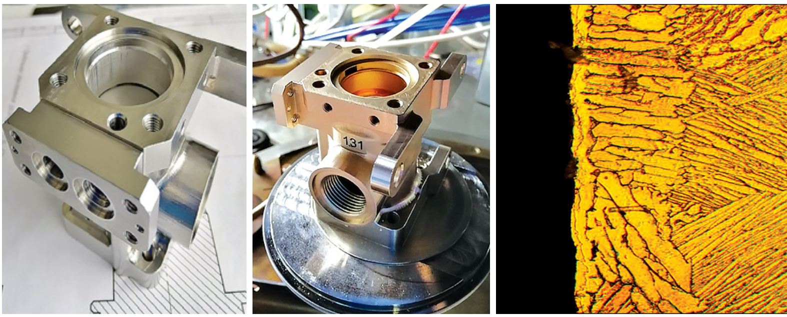


Fig. 3.33 Housing from DIN 3.7165 titanium alloy. The structure of the nitrided layer on the inner cylindrical part of the housing surface

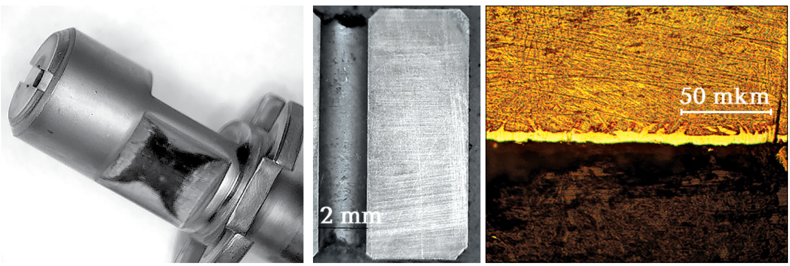


Fig. 3.34 Slide valve. Structure of nitrided layers on slide valve surface

Generalized parameters for the implementation of the of plasma precision nitriding Avinit N method are presented in Table 3.10.

Table 3.10 Generalized parameters for the implementation of the of plasma precision nitriding Avinit N method

Method's parameters	34NiCrMoV14-5	DIN 1.773	24CrMoV5-5	Alloy DIN 3.7165
Heating temperature in the initiated gas-discharge plasma, T °C	400 ± 5	400±5	400±5	400±5
Dwell time, min.	20±10	20±10	20±10	20±10
Negative purification potential, V	600±5	600±5	600±5	600±5
Isothermal holding time, hr.	1.5	1.5	1.5	1.5
Nitrogen pressure, mm·Hg·10^{-3}	1.5±0.1	1.5±0.1	1.5±0.1	1.5±0.1
Displacement potential at nitriding, V	600±5	600±5	600±5	600±5
Nitriding temperature, T °C	530±5	530±5	530±5	700±5
Nitriding time, h	2	2	2	4

Comparative characteristics of traditional nitriding and Avinit N plasma precision nitriding processes are presented in Table 3.11 for widely used commercially available steels 34NiCrMoV14-5, DIN 1.773, 24CrMoV5-5 and titanium alloy DIN 3.7165.

Table 3.11 Comparative characteristics of traditional nitriding and Avinit N plasma precision nitriding processes

Material	Process parameters, properties	Traditional processes of ion application	Precision plasma nitriding
1	2	3	4
34NiCrMoV14-5	Time for obtaining of hardened layer with thickness 0.2...0.3 mm, hr.	16	2
	Process temperature, °C	500–600	530
	The nitrided layer depth, mm		0.25
	Hardness of nitrided layer, HV		830
	Base hardness, HRC		37–39
	Geometrical dimensions characteristics with accuracy 1–2 μm		unchanged

Continuation of Table 3.11

1	2	3	4
DIN 1.773	Time for obtaining of hardened layer with thickness 0.2...0.3 mm, hr.	16	2
	Process temperature, °C	500–600	530
	The nitrided layer depth, mm		0.25
	Hardness of nitrided layer, HV		790
	Base hardness, HRC		36–40
	Geometrical dimensions characteristics with accuracy 1–2 μm		unchanged
24CrMoV5-5	Time for obtaining of hardened layer with thickness 0.2...0.3 mm, hr.	20	2
	Process temperature, °C	500–600	530
	The nitrided layer depth, mm		0.3
	Hardness of nitrided layer, HV		970
	Base hardness, HRC		38
	Geometrical dimensions characteristics with accuracy 1—2 μm		unchanged
Titanium alloy	Process temperature, °C	700–800	700
	Layer thickness with hardness ≥600 HV, mm	0.01	0.05
	Nitriding time, hr.	15	4
	The nitrided layer depth, mm		0.05
	Hardness of nitrided layer, HV		950
	Base hardness, HRC		37–39
	Geometrical dimensions characteristics with accuracy 1–2 μm		unchanged

According to experimental data, obtaining a layer with a uniform surface structure in the diffusion saturation of nitrogen under the action of ions of a gas mixture of argon and nitrogen, created by a gas plasma generator, increasing the hardness of parts from steels and alloys with minimal distortion and preserving the original geometric dimensions of the products is achieved by plasma precision Avinit N nitriding with obtaining a uniformly strengthened layer in the absence of the brittle layer of iron nitrides formation.

The temperature of the parts in the nitriding process for steels is 500–600 °C.

3.4.2 Contact fatigue strength

An important characteristic of materials is the ability to withstand the fatigue fracture of work surfaces, which is characterized by such a value as the contact strength of the material. In the flow charts of parts manufacturing for increasing the contact strength of materials, chemical-thermal methods of surface hardening are widely used — mainly cementation methods, which have significant drawbacks.

In the case of cementation, hardening occurs with the simultaneous accumulation of energy at the interface of the grains, which subsequently lead to the growth of microcracks.

In addition, the release of energy during the contact interaction of the surfaces leads to their heating and destruction in the cemented layer of carbides, resulting in a decrease in contact strength.

The application of traditional methods of gas nitriding requires, often, very time-consuming, complex operations of high-precision mechanical grinding, as, for example, in the manufacture of parts of high-precision gears. In this case, due to the formation of a brittle nitride layer, it is sometimes necessary to grind it to a depth of 0.1 mm, which can be a significant part of the entire reinforced layer and, as a consequence, a significant deterioration of mechanical properties.

Widely used in the industrial production of JSC "FED" methods of plasma precision nitriding Avinit N, have significant differences and advantages over traditional rough methods of gas nitriding.

In the work [56] using the acoustic emission method, we conducted comparative tribotechnical tests for contact fatigue strength during rolling friction with the slip of surfaces, hardened by cementation and Avinit N plasma nitriding, with contact loads $\sigma_{max}\approx 1000$ MPa.

The average results of tribotechnical parameters when testing pairs of samples for fatigue strength are given in Table 3.12.

Table 3.12 The average results of tribotechnical parameters in accelerated tests

Strengthening of samples		Coefficient of friction/average surface temperature, °C		The number of cycles before the onset of fatigue failure
Leading sample	Driven sample	At the beginning of the test	In the end tested	
Cementation	Cementation	0.054/43	0.045/45	53812
Nitriding	Nitriding	0.08/50	0.063/52	97875

Measurement of the depth of damage on the surface of the samples using an interferometer MII-4U4.2 showed that for cemented surfaces, it, depending on the diameter of the damage, is in the range from 0.01 to 0.027 mm for nitrided surfaces, the depth of damage did not exceed 0.003 mm. In the cemented layer after 960000 cycles there is a significant decrease in microhardness, apparently due to the accumulation in the surface layer of fatigue defects of the microstructural level and as a consequence, increase the integral value of wear.

For samples with nitriding, the values of microhardness remain almost at the same level, which is a confirmation of the amorphization of the surface layer, and, consequently, the accumulation of fatigue defects to a much lesser extent.

Thus, test results based on 1000000 cycles (rolling with slip with contact loads $\sigma_{max} = 1140$ MPa, characteristic of medium-loaded surfaces) indicated that integral multi-cycle resistance to fatigue wear (destruction) of samples, hardened by nitriding of Avinit N with layer depth of 0.25 mm, more than by 10 times higher than samples hardened with cementation with a layer depth of 1.2 mm.

Advantages of plasma precision nitriding Avinit N

The main advantages of the method are the significant intensification of the nitriding process, obtaining a uniformly strengthened nitrided layer, the absence of a fragile layer without distortion of the parts while preserving the original geometric dimensions. After Avinit plasma precision nitriding, the drawing dimensions are retained. The absence of a fragile layer of the surface layer allows to avoid finishing grinding after nitriding, and receive the nitriding operation "to the size".

The use of the method intensifies the process of creating a nitrided layer by 3–5 times in comparison with the treatment by the traditional method of ion nitriding in glowing discharges and − by 5–30 times when compared with gas nitriding. Hardness and durability of the parts increases by obtaining a uniformly reinforced nitrided layer. For some precision complex-geometry parts that do not allow for 1–2 microns out-of-flat condition after nitriding, and for which machining with high precision grinding of hard nitrided surfaces is not possible, Avinit N "to the size" plasma nitriding is the only way to obtain the ready-to-use product.

Compared to widely used nitriding methods, the plasma nitriding method has the following main advantages:

- absence of parts deformation (distortion) after processing, reduction in parts deformation which allows to eliminate the residual grinding;
- constant quality of processing with minimal dispersion of properties from part to part and from melting charge to melting charge;
- no pollution of the environment;
- the process is environmentally friendly, does not use hydrogen, ammonia, and hydrogen-containing compounds;
- raising the production culture;
- reducing the processing cost.

The benefits of Avinit N plasma nitriding also manifest themselves in a significant reduction in major production costs.

Compared to gas nitriding in furnaces, Avinit N plasma nitriding provides:

- reducing processing duration by 10–50 times, both due to reducing processing time by 85 % and eliminating the residual high-precision refinement;
- absence of ammonia and hydrogen-containing compounds in the working gases, reducing the working gases consumption by 80 %;
- reduction of electricity consumption by 70–75 %;
- reduction of parts deformation while excluding finishing grinding;
- improvement of production sanitary and hygienic conditions;
- full compliance of technology with all modern requirements for environmental protection and environmental safety.

The results of tribological tests showed a significant advantage of samples with nitriding reinforcement Avinit N compared to cementation. Samples hardened by Avinit N technology have higher wear resistance, taking into account the fatigue mechanism of surface fracture.

This makes it possible to make extensive use of Avinit N plasma nitriding technologies, instead of cementation, to increase the contact strength of the surface in the manufacture of critical parts.

Plasma precision nitriding processes eliminate the disadvantages of traditional industrial nitriding processes, increase the performance of parts, expand the range of materials to be processed.

Plasma precision nitriding Avinit N is successfully used in mass production instead of traditional ionic nitriding, liquid and gaseous nitriding.

3.4.3 Development of duplex technologies Avinit

Even more significant results in increasing the contact long-term strength can be expected when applying antifriction coatings to the nitrided surface.

When using metal surface coatings (copper, palladium), such antifriction coatings have limited performance due to abrasive wear. In the 1990s, tungsten carbide-based superhard ceramic coatings began to be introduced in the West.

JSC "FED" has developed complex Avinit technologies that allow to perform multilayer antifriction coatings [50, 66], which consist of nanolayers of titanium nitrides, aluminum nitrides and titanium. The developed technologies for applying Avinit multifunctional coatings have shown the high antifriction properties of Avinit coatings and high efficiency in many industrial applications when applied to critical parts in aggregate construction, transport engineering and energy engineering.

On the basis of the carried out scientific and experimental researches on a choice of materials of nanocoatings of Avinit and technologies of their reception their efficiency in friction pairs in aggregate construction is experimentally proved.

Strengthening of turbocharger blades of gas turbine engines with wear-resistant Avinit coatings (Fig. 3.35)

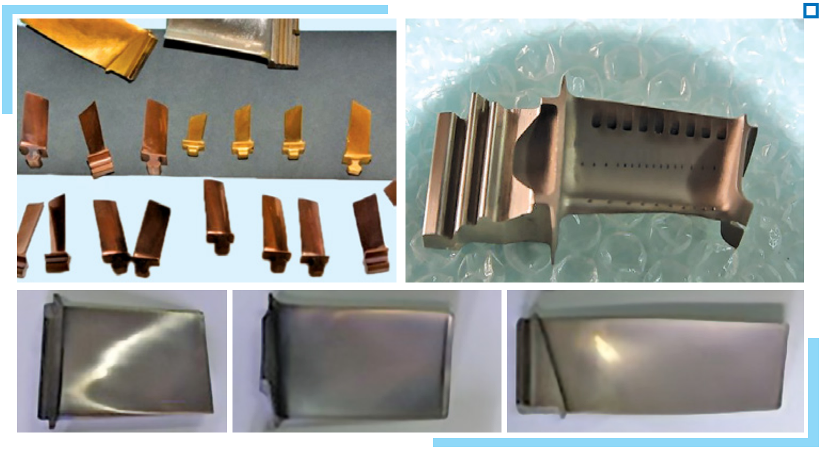


Fig. 3.35 Coatings Avinit C 350 based on (Ti-Al-N) and Avinit D 100 based on Ti-C

Introduction into the production of pumps and regulators of fuel supply units and control of aircraft engines of developed technologies for applying nanolayer low-temperature (≤150–200 °C) coatings Avinit provides high reliability of serial and new designs of units and increases installation and maintenance life by 5–20 times (Fig. 3.36).

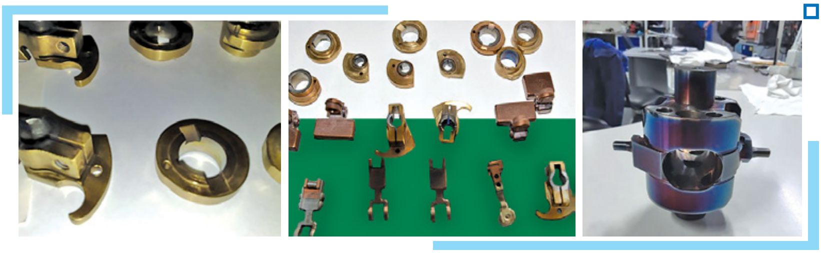


Fig. 3.36 Details of serial aviation hydraulic units with Avinit nanocoatings

The use of spools with Avinit coatings in hydraulic units increases their service life from 200 to 2000 hours.

However, obtaining high-quality Avinit coatings on surfaces nitrided by traditional methods is associated with great difficulties, mainly due to the presence on the nitrided surface of a fairly thick layer of brittle ε-phase and significant distortion of parts. To bring the dimensions to the requirements of the technical documentation after nitriding requires further grinding and polishing of work surfaces, which significantly reduces the contact strength of the surface and increases the complexity of manufacturing products. The mechanical parameters of this thick brittle layer are poorly compatible with the coating and as it turned out, further vacuum-plasma application of high-quality wear-resistant coatings is possible only with the complete removal of these fairly thick layers from the coated work surfaces. For complex surfaces, this becomes quite difficult and time-consuming, and sometimes simply unsolvable technical problem.

Our research has shown that the absence of a brittle surface layer after plasma nitriding allows further application of high-quality functional coatings Avinit without any mechanical improvements of the nitrided surface (Fig. 3.37). In this regard, of undoubted interest is the development of duplex technology that combines plasma nitriding of the finished high-precision parts with the subsequent application of superhard antifriction coatings Avinit in a single process, which will avoid many difficulties, associated with the use of gas nitriding details.

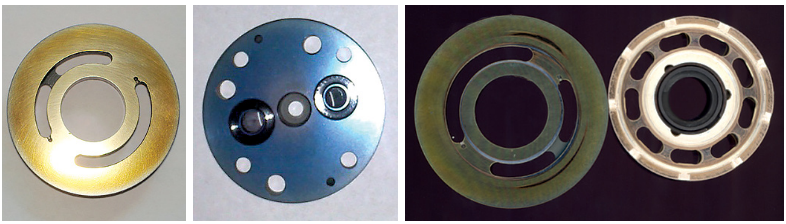


Fig. 3.37 Spools of hydraulic units with Avinit nanocoatings

We have developed complex duplex technologies Avinit [17, 67, 68], including plasma nitriding Avinit and ion-plasma application of wear-resistant coatings Avinit in a single process. Avinit duplex processes are performed on an Avinit vacuum-plasma cluster.

First, plasma precision nitriding of Avinit N parts is performed. The parameters of the process and its duration are determined on the basis of the results of previous experiments. Through appropriate electronic switching, the Avinit installation is switched to coating mode.

The temperature of the parts is reduced to the parameters required for vacuum-plasma coating of Avinit C according to the established technology.

After the coating process, careful control was carried out for compliance of the thickness and microhardness of the applied coatings with the requirements of the technical documentation by metallographic and profilographic analysis of the sections of the witness samples.

Changing the process parameters (pressure, temperature, time, and discharge voltage) allows to provide the specified properties of nitrided surfaces and to produce high-precision parts.

Examples of duplex technologies Avinit N [17]

1. Full-size high-precision gear

In accordance with the developed technological regulations, plasma precision nitriding of Avinit N and subsequent application of Avinit antifriction coating of a full-size high-precision gear with Avinit duplex technology were performed. The hardness of the nitrided layer is given in Table 3.13.

Table 3.13 The hardness of the nitrided layer

№	Distance from the edge, mm	Hardness, H_{μ}, in brackets HRC
1	0.05	603, 644 (57.5), 633 (57)
2	0.1	612 (56), 557 (53), 584 (54)
3	0.15	532, 532 (51), 516
4	0.20	524, 524 (50.7)
5	0.25	501 (49), 501, 494 (49)
6	0.30	453 (45.6)
7	0.35	466, 453 (46)
8	0.40	401 (41), 391 (39.8)
9	0.45	376, 390

On the JEOL 6360LA electron microscope, the Avinit coating on the surface of the nitrided layer was studied by the PCMA method.

The research results are presented in Fig. 3.38.

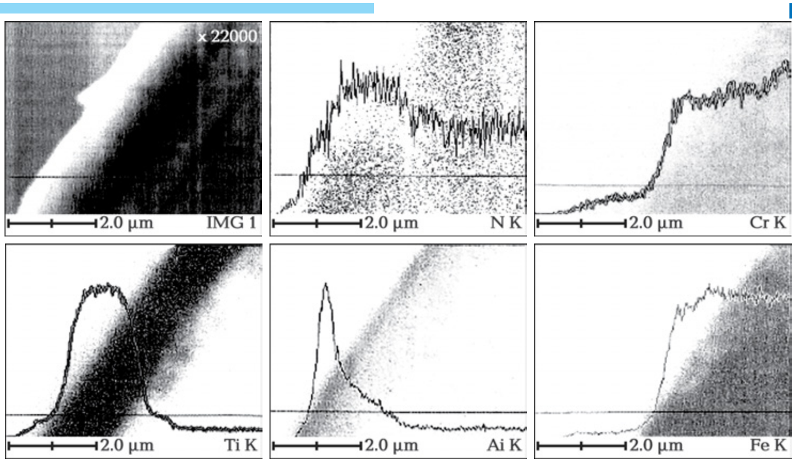


Fig. 3.38 Appearance of Avinit coating in line analysis mode. The coating thickness is 1.5 µm. The microhardness of the coating was 2500...3000 HV

It is established that changes in geometry after plasma precision nitriding of Avinit N and Avinit coating by duplex technology are not observed. Avinit antifriction coating with a thickness of 1.5 μm does not distort the geometry of the tooth profiles. All parameters of the gear ring, made by duplex technology Avinit, meet the requirements of the technical documentation (Fig. 3.39).

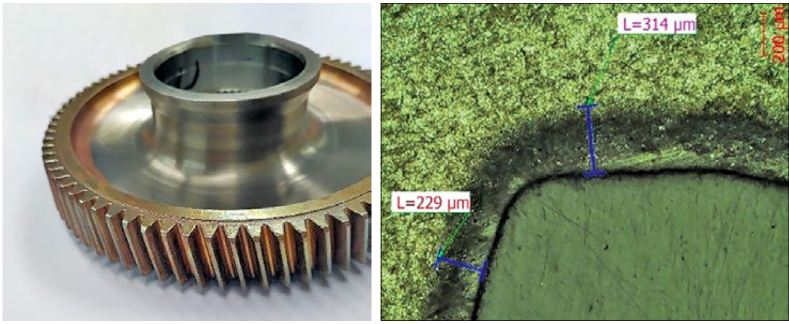


Fig. 3.39 Full-size high-precision gear, made using duplex technology Avinit. Depth of the nitrided layer 0.3 mm. Microhardness $H_{\mu}=730–930$. Avinit coating layer thickness 1-2- μm, $H_{\mu}=3000$

A pair of experimental gears, reinforced by duplex technology Avinit, successfully passed field tests as part of the gearbox of the AI-450M engine on the hydraulic brake stand of SE "Ivchenko-Progress" in accordance with the program of equivalent cyclic tests Avinit [68].

No changes in the geometry of the wheels teeth and the wear of the coating Avinit after testing in the engine gearbox were detected.

2. Freewheel clutch separator

Avinit duplex technology has been used to improve the performance of freewheel clutch separator of the main reducer of the helicopter.

The works [17, 67] is devoted to the development of Avinit duplex technology on complex parts to increase the wear resistance of the separator by plasma precision nitriding Avinit N and subsequent application of Avinit antifriction coating on friction surfaces and receiving power loads.

Plasma chemical-thermal treatment and application of antifriction coating by duplex technology Avinit was carried out on the separators.

The results of measurements of hardness, depth of the nitrided layer and the thickness of the antifriction coating Avinit are presented in Table 3.14.

Measurements of microhardness along the depth of the nitrided layer show that the nitrided layer is evenly distributed over the entire surface of the separator, the effective depth of the nitrided layer is 0.2 mm.

Table 3.14 Characteristics of the nitrided layer and the thickness of the coating Avinit

The name of the zone	Hardness			Depth of the nitrided layer, mm	Thickness of coating Avinit, mm
	Of coating Avinit, HV 0.05	Of nitrided surface, HRN 15	Of the base, HRC		
End groove	1042...1124	92.0...93	35	0.2	0.001...0.002
∅ 52		91		0.2	0.001
TD norms		≥83	27.0...38.5	0.2	0.001...0.002

The thickness of the coating layer Avinit is uniform and is 1–2 µm, $H_{\mu} = 3000$. The quality of adhesion of the coating with the part is satisfactory, no signs of peeling are detected.

All parameters of the experimental batch of separators made by duplex technology Avinit, fully meet the technical, metallurgical and metallographic requirements of CD (thickness and microstructure of the nitrided layer, lack of fragility, etc., thickness and hardness of the coating, preservation of geometric parameters with an accuracy of 0.5 µm) (Fig. 3.40, 3.41).

Fig. 3.40 Freewheel clutch separator. Plasma nitriding of Avinit N + Avinit C coating

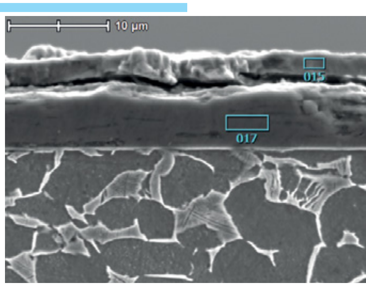


Fig. 3.41 The structure of the nitrided layer. Depth of the nitrided layer 0.2 mm. Microhardness $H_{\mu} = 730–830$. Avinit coating layer thickness 1-2- µm, $H_{\mu} = 3000$

The Avinit-coated separator has been successfully tested as part of the main gearbox on a full-scale stand.

Bench tests of the separator, which is a part of the main gearbox of the aircraft engine, indicated that Avinit plasma nitriding in combination with the subsequent deposition of Avinit C superhard coatings provides no fretting wear of the working surfaces, characteristic of commercial separators.

Thus, the developed duplex technology of Avinit by the method of plasma precision nitriding of Avinit N and the subsequent application of Avinit C coating on work surfaces ensures the application of tightly bonded, high-quality multifunctional antifriction coatings of Avinit C on heavy-duty parts (Fig. 3.42).

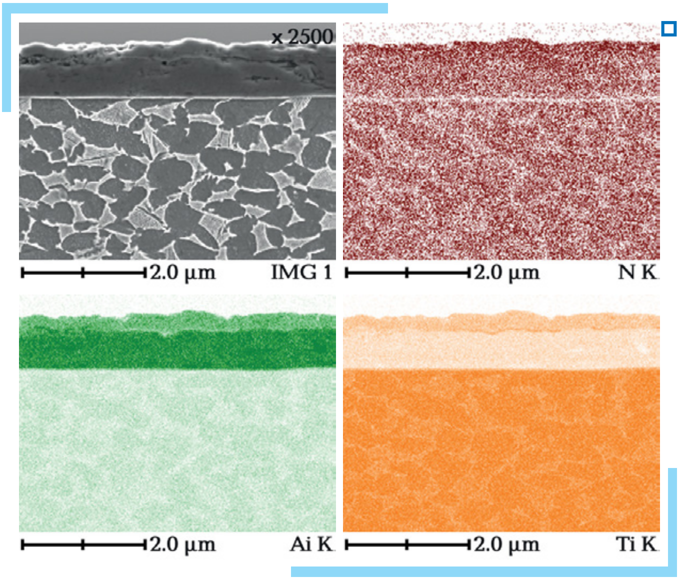


Fig. 3.42 Appearance of Ti-Al-N coating (cross section) in mapping mode

4

Development of industrial research technologies for Avinit coatings

4.1 The choice of coatings to improve the reliability of thermal equipment of turbines

Summarizing the results of studies on corrosion, erosion, friction, adhesion, roughness of vacuum-plasma coatings described in Section 3, it is possible to formulate that such coatings in relation to the tasks of turbine construction are characterized by the following parameters:

- coatings have high microhardness, excellent adhesion to the base, low coefficients of friction and good resistance to abrasions during friction;
- erosion resistance of coatings is 30 % higher than that of widely used stellite, and unlike stellite, no finishing is required;
- resistance of coated products to corrosion can be compared with the resistance of the best corrosion-resistant materials, such as solid titanium;
- high efficiency of coatings is maintained under the influence of tensile stresses of the operating level in a corrosive environment;
- process of coating formation does not have a negative impact on the mechanical properties and structure of structural materials;
- endurance limit of coated steel increases by 15–25 %.

Avinit wear-resistant high-hard coatings applied at low temperatures provide good adhesion to substrate materials without a significant reduction in steel strength (<200 °C) and without deterioration of the purity class of the original surface.

4.2 Coatings to increase the service life of steam distribution and steam control mechanisms of turbines

Since 2005, together with OJSC "Turboatom" successfully work on the development and commercialization of the latest technologies for the application of wear-resistant antifriction coatings using nanotechnology to increase the life of various critical elements of steam and nuclear turbines.

The determining factor of wear in most cases is the chemical composition of the friction surfaces. There are a number of units and mechanisms in the friction units of which, along with the processes of mechanical friction, complex physicochemical processes occur due to the interaction of friction surfaces under the action of aggressive environments.

Such very loaded friction pairs are parts and units of mechanisms of steam regulation and steam distribution of turbines (cams and rollers, sliding bearings, saddles of high-pressure valves, a rod, etc.) which work in the conditions of corrosion and erosion influences and high specific loadings (to 200 kg/mm^2 and above), part of the nodes — in conditions of high contact loads.

A significant number of studies have been performed to determine the optimal compositions and constructions of coatings based on Cr, Mo, Ti and their nitrides.

The application of coatings on the surface with a purity of treatment up to class 13 roughness required solving the problem of purification of the plasma flow from the "drip component".

As shown by experimental results, the use of an advanced rectilinear separator provides the formation of plasma streams purified from microparticles of cathode material, which allows to deposit the coating on the surface of V 11–13 class almost without changing the purity class of the surface. Comparison of the roughness of the substrate and the coating showed that the coating has a slight increase in roughness (Table 4.1), which practically does not go beyond one class of surface roughness.

Table 4.1 shows the data on the microhardness and roughness of the studied coatings on samples of steel DIN 1.773, polished to 10–12 class.

Table 4.1 Characteristics of the studied samples with multilayer coatings

№	Coatings	Thickness, µm	Microhardness, $HV_{(50g)}$, MPa	Initial roughness, R_a, µm	Roughness, R_a, µm
1	Ti	8–10	11000	0.026	0.040
2	Mo	8–10	10000	0.017	0.026
3	Ti-TiN	8–10	15000–20000	0.018	0.036
4	Mo-MoN	8–10	18000–22000	0.016	0.025

According to the metallographic studies of the samples after coating, the spent modes provide the formation of high-quality coated coatings. Coating does not reduce the hardness of the original surface.

The microhardness of the coatings has a value in the range from 15000–20000 MPa. The coatings have good adhesion to the substrate material. There were no cases of peeling of coatings when applying a grid of scratches.

The most promising materials for wear-resistant coatings for a set of properties — corrosion, erosion, friction, adhesion, roughness in the conditions of operation of the responsible elements of the mechanisms of steam distribution and steam regulation of steam and nuclear turbines are molybdenum nitride and titanium nitride.

To eliminate the adhesion of parts working in pairs with each other, according to vacuum-plasma technologies developed by STC "Nanotechnology", as shown by numerous studies, it is advisable to apply coatings of different chemical composition:

- on concave surfaces — titanium nitride;
- on convex surfaces — molybdenum nitride.

After numerous technological tests of the developed coatings on the parts of JSC "Turboatom", it was decided to use these coatings in the friction pairs of the responsible parts of the turbines in serial production.

OJSC "Turboatom" and STC "Nanotechnology" were issued and included in the design documentation Technical conditions for vacuum-plasma application of wear-resistant coatings designed to protect parts operating in hinged joints and sliding bearings of turbines prone to corrosion and corrosion conditions of dry friction in air and friction in humid steam and water environments for temperatures up to 320 °C.

The processes of vacuum-plasma application of wear-resistant coatings are carried out on the Avinit installation in vacuum with a residual pressure not exceeding $5{\cdot}10^{-5}$ mm Hg.

1. Before carrying out processes of drawing wear-resistant coverings careful preparation of a surface of the details which are exposed to vacuum-plasma drawing of wear-resistant coverings of titanium nitride and molybdenum nitride on Avinit technologies is carried out:

- removal of traces of scale and sandblasting;
- polishing of work surfaces with scratches and abrasions;
- work surfaces must not have corrosion damage, deep scratches, sinks, cracks, metal rubs. Traces of paint, oil and other mechanical contaminants are not allowed;
- working edges must not have dents, chips, burrs and burns. Traces of contamination by foreign inclusions, traces of corrosion and scale are not allowed.

The duration of storage of parts after the operation of surface preparation before coating should not exceed 12 hours.

2. Working details are installed in the special technological devices protecting parts of details which, according to the technical documentation, should not be exposed to a covering. All technological equipment must be made of steel with vacuum-plasma coatings of titanium nitride or molybdenum nitride.

3. Vacuum-plasma application of wear-resistant coatings of titanium nitride and molybdenum nitride on the part in accordance with TU.

4. Together with details the covering of samples-witnesses on which control of thickness and hardness of coverings is carried out is carried out.

Types of control

1. The stability of the processes of surface cleaning of parts and application of functional coatings is controlled by the technological parameters of the process by comparing them with the requirements of technological documentation.

2. After coating, 100 % control of coated parts is carried out for the absence of visible coating defects, such as: peeling ("shooting"), damage by microarcs, especially sharp edges, uneven coating, the presence of uncovered areas.

The presence of individual drops and shells (recesses) is allowed.

Local chipping of the coating on non-working surfaces of parts is allowed.

3. The presence of chips or dents is not allowed. Swelling and peeling of the coating is not allowed. Parts with cracks and dents in the coating are missing.

4. Metallographic and profilographic analyzes of sections of witness samples shall check the compliance of the thickness and microhardness of the applied coatings with the requirements of the technical documentation.

OJSC "Turboatom" and STC "Nanotechnology" issued Technical Conditions for vacuum-plasma coating of wear-resistant coatings on turbine parts for operation in friction units with specific pressure on contact surfaces up to 200 kg/mm^2 in conditions of dry friction in air and friction under wet environments for temperatures up to 320 °C. TU are included in the design documentation.

Given the extensive positive experience in the industrial implementation of the latest technologies for wear-resistant antifriction coatings using nanotechnology to increase the life of parts and components of steam control and steam distribution of turbines and industrial operation of such elements with coatings at temperatures up to 320 °C, it was decided to expand the scope of developed protective coatings and technologies for their application to increase the life of power equipment and to protect various parts operating under high pressure, static and dynamic loads in abrasive and chemically aggressive environments and able to maintain performance at temperatures up to 560...580 °C.

To this end, scientific and technological developments have been made to study coating systems for protection against burrs and erosion of parts of steam distribution bodies operating at temperatures up to 580 °C.

Tests of coatings on stability at a temperature of 565 °C and on heat change − 565 °C with cooling in air to 20 °C are carried out.

Based on the obtained positive results, the Technical Conditions for vacuum-plasma coating of wear-resistant coatings on turbine parts manufactured by OJSC "Turboatom" for work in friction units with specific pressure on contact surfaces up to 200 kg/mm^2 in conditions of dry friction in air have been developed and agreed. and friction in humid and water environments, as well as for work in the seals of the shut-off organs in the steam, humid and water environments at temperatures up to 580 °C.

Adaptation of technologies of formation of multicomponent multilayer protective coverings possessing high erosion and corrosion resistance to requirements of technical specifications for realization in the conditions of serial production of vacuum-plasma technologies for protection of details of bodies of steam distribution and regulation of steam turbines is carried out.

4.3 Avinit duplex technologies

Standard technology for the production of parts designed to work in hinged joints and plain bearings of turbines, which are made of steels such as DIN 1.773, 24CrMoV5-5 etc., includes pre-nitriding and subsequent vacuum-plasma coating of wear-resistant nitride coatings in accordance with the technical conditions of the TU.

Nitriding of working surfaces is important for increase of contact durability of details.

Preliminary nitriding of working parts to a hardness of at least 550 HV30 in the factory is carried out by the traditional method of gasnitriding.

The main disadvantages of this technology are the presence on the nitrided gas nitriding surface of the solid brittle ε-phase and significant distortion of the parts.

To bring the dimensions to the requirements of the technical documentation after nitriding requires further grinding and polishing of work surfaces, which significantly reduces the contact strength of the surface and increases the complexity of manufacturing products.

The mechanical parameters of this thick brittle layer are poorly compatible with the coating and as it turned out, further vacuum-plasma application of high-quality wear-resistant coatings in accordance with TU is possible only with complete removal of solid brittle ε-phase from nitrogenous parts.

We have developed the process of plasma precision nitriding Avinit N [17, 55, 64, 65], based on the diffusion saturation of the surface layer of steel parts with nitrogen in high-density low-temperature non-equilibrium plasma of nitrogen and argon, provides no deformation (distortion) geometric dimensions after nitriding with an accuracy of 1–2 μm, the absence of a brittle surface layer, which avoids the finishing grinding of parts and get the operation of precision nitriding "in size".

Plasma precision nitriding processes eliminate the shortcomings of industrial nitriding processes (traditional ionic nitriding, liquid and gaseous nitriding), significantly reduce their duration, provide a stable quality microstructure of the surface layers of parts, eliminate their fragility, increase the performance properties of parts.

Studies have shown that the absence of a brittle surface layer after plasma nitriding allows for further application of high-quality functional coatings without any mechanical improvements of the nitrided surface.

Paragraph 3.4.3 presents the results of research to create a duplex process that combines plasma nitriding Avinit final finished high-precision parts with subsequent vacuum-plasma application of functional superhard coatings Avinit.

Based on these studies, complex Avinit technologies were developed, including plasma nitriding and ion-plasma coating of wear-resistant coatings based on titanium nitride and molybdenum nitride in a single process, for the manufacture of parts of steam distribution mechanisms and steam control of steam and atomic turbines.

Avinit duplex technologies allow to ensure the application of strong, high-quality coatings of titanium nitride and molybdenum nitride on the parts of OJSC "Turboatom" in accordance with the Technical Specification, while significantly improving the economic performance of the parts.

Together with OJSC "Turboatom" developed and agreed Technical Conditions – nitriding and ion-plasma coating of wear-resistant coatings based on titanium nitride and molybdenum nitride on parts of complex technologies Avinit.

After the coating process, a careful control is carried out on the compliance of the thickness and microhardness of the applied coatings to the requirements of the technical documentation by metallographic and profilographic analyzes of the sections of the witness samples.

The developed complex duplex technologies of Avinit are introduced into the corresponding technical normative documentation of production of details of mechanisms of steam distribution and steam regulation of steam and atomic turbines made by OJSC "Turboatom". Depth of the nitrided layer is 0.2 mm, thickness of a coating is 14 microns (Fig. 4.1).

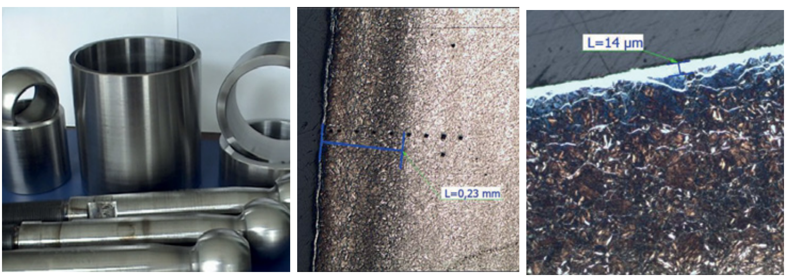


Fig. 4.1 Duplex technology of Avinit on turbine steam distribution elements

4.4 Vacuum-plasma multilayer protective coatings for turbines blades

We have developed [67, 69, 70] vacuum-plasma methods of multistage surface pre-treatment, which, as the research results indicate, when combined with optimization of the multilayer structure allow to obtain high-quality firmly bonded coatings.

The developed process, in which the deposition of a protective multilayer erosion-resistant coating in vacuum-arc deposition is performed through the multistage ion-plasma surface cleaning and formation of optimized multilayer structure of layers with predetermined repeated periods, and the thicknesses of the individual layers subject to the set programs which ensure software-synchronized pressure regulators control of inert and reaction gases and electrical process parameters.

When designing multilayer coatings it is expedient to create soft interlayers prior to deposition of hard coatings. Soft layer improves adhesion of the coating with substrate material, ensures the presence of a large positive gradient of mechanical properties in the coating, which is a good prerequisite for normal working in conditions of friction and shock loads. These layers prevent the propagation of cracks arising in the course of operation.

This principle is used to form an optimized multilayer coating design.

The experiments were performed on the witness specimens made of steel 1.4021 Ge and titanium alloy DIN 3.7165, which are used for the manufacture of blades, with the purpose of studying the different structures of coating (total coating thickness, thickness of layers, their sequence, the parameters of the stabilization annealing) with regard to the adhesion and quality of coatings, to invent optimal design of multilayer protective coating (Tables 4.2, 4.3).

The layer thickness control is performed using pre-calibrated meter thickness for FTC-2800 that allows to measure the coating growth rate from 0.01 A/sec. Applying layered coatings is carried out by setting pre-set values for nitrogen pressure that is ensured by the software-controlled nitrogen pressure regulators. The creation of the necessary cycle schemes to obtain set recurrence intervals and thickness of the individual layers is provided by software-controlled pressure regulators of nitrogen pressure from the thickness meter FTC-2800. To measure the temperature of the components the pyrometer Raytek is used.

Metallographic examination and determination of material parameters (coating thickness, uniformity, and defect structure of the material) was performed using microscopes MMR-4 and Tesa Visio 300 gL. The coatings microhardness was measured using the PMT-3 and BUEHLER microhardness testers at a load of 50 g.

The coatings adhesion was measured using Revetest Scratch Tester (RST) scratch meter.

Table 4.2 Experiments for the choice of multilayer protective coating design

№	Substrate material	№	First layer Ti			Second layer Ti-TiN							Common layer characteristics		
			Process parameters		First layer characteristics	Process parameters			Second layer characteristics Ti-TiN						
			Current I_p (Ti), A	Voltage U_c, V	General thickness of the first layer, μm	Current I_p (Ti), A	Voltage U_c, V	Nitrogen pressure P_{N2}, Pa	Layer thickness Ti, nm	Layer thickness TiN, nm	Recurrence period, nm	Total thickness of the second layer, μm	Adhesion	Total thickness, μm	Microhardness, MPa
1	1.4021 Ge	1.1	90	120	5 Ti	90	120	$2.0 \cdot 10^{-1}$	–	–	–	10 Ti	Good	15	20.000
		1.2	90	90	2 Ti	100	90	$2.0 \cdot 10^{-1}$	5	10	15	15 Ti-TiN	Good	17	19.000
		1.3	120	100	3 Ti	100	100	$2.0 \cdot 10^{-1}$	3	15	20	12 Ti-TiN	Good	15	19.000
		1.4	120	120	3 Ti	120	120	$2.0 \cdot 10^{-1}$	5	10	15	10 Ti-TiN	Good	13	19.000
		1.5	120	120	5 Ti	120	120	$2.0 \cdot 10^{-1}$	2	8	10	15 Ti-TiN	Very good	20	18.000
		1.6	100	100	3 Ti	100	120	$2.0 \cdot 10^{-1}$	10	10	20	17 Ti-TiN	Good	20	17.000
		1.7	120	140	5 Ti	120	140	$2.0 \cdot 10^{-1}$	10	20	20	20 Ti-TiN	Good	25	19.000
2	Titanium alloy DIN 3.7165	2.1	90	120	5 Ti	100	120	$2.0 \cdot 10^{-1}$	–	–	–	10 Ti	Good	15	20.000
		2.2	100	100	5 Ti	100	100	$2.0 \cdot 10^{-1}$	10	10	10	15 Ti-TiN	Good	20	19.000
		2.3	120	120	5 Ti	120	120	$2.0 \cdot 10^{-1}$	2	8	10	15 Ti-TiN	Very good	20	18.000
		2.4	100	100	5 Ti	120	120	$2.0 \cdot 10^{-1}$	15	20	20	15 Ti-TiN	Good	20	17.000

Table 4.2 provides the results of the experiments performed for the choice of multilayer construction of the protective coating.

As can be seen from the Table 4.2 the best design of the multilayer protective coating in terms of quality, with regard to adhesion, is the construction provided in para 1.5 of Table 4.2, namely:

erosion-resistant coating which contains a metal layer of titanium and layers of titanium nitrides, is a multi-layer structure, in which:

- a primary coating layer made of Ti with a thickness of 3–5 μm;
- the second layer is made as a component of the layers (Ti-TiN) with recurrence period of 10 nm and the thickness of the individual layers of 2 nm and 8 nm respectively, with a thickness of 10–15 μm.

A process of multi-stage ion-plasma surface cleaning is performed as follows

Stage 1 – treatment in a glow discharge plasma of inert argon gas. Due to low values of plasma density and ion flux density on the treated surface, the speed of cleaning (sputtering of the surface layers) in the glow discharge plasma is much lower than the speed of cleaning in the plasma of electric arc discharge.

Higher voltage (600 V) required for combustion of the glow discharge in such conditions can cause the microarcs appearance on the surface contamination and the purity class deterioration. It is not possible to completely suppress the process of the microarcs emergence, even despite the presence of well-designed system protecting against microarcs.

So the glow discharge treatment was used for the pre-degassing and "activation" of fairly clean surfaces with a very smooth potential increase.

The results of these studies indicate that the duration of surface treatment by argon ions in the argon glow discharge plasma should not exceed 30 min.

The plasma parameters (ion current, ion density, current-voltage characteristics, spectral characteristics) during the ion-plasma treatment was continuously monitored and archived with plasma meter "PlasmaMeter" and "PlasmaSpectr" spectrometer.

Then ion-plasma processing is carried out in high-density argon gas plasma which is generated by the gas plasma generator of Avinit installation.

Stage 2 – processing high-density plasma of two-stage vacuum-arc discharge inert argon gas, as a powerful plasma source gas of plasma source, created by gas plasma generator for high-efficiency ion-beam treatment that provides strong adhesion of the coating to the substrate material and deposition of high-quality functional coatings.

For the implementation of the gas plasma mode at the output of the electric arc source optically opaque partition is installed which hinders the electric field effect, which is basically a louvered screen to prevent the entry of metal ions onto a part.

High-density gas plasma is excited between the cathode of the electric arc source, in proximity to which the screen is installed, and the plasma duct of the opposite electric arc source that is isolated from the vacuum chamber.

The cathode of such a discharge is a cathode of the arc discharge, isolated from the chamber with a special separator, which prevents the metal flow, but it allows to create a high-density gas plasma in the chamber.

Using the probe monitoring system of plasma technological processes plasma meter "PlasmaMeter" comparative probe measurements of plasma parameters of various plasma sources of the upgraded installation (glow, double arc discharges) were carried out. They indicated that the ratio of flows of ions and neutral atoms for dual arc discharge plasma generates approximately 300–1000 times more intense ion flux in comparison with the glow discharge.

This much denser plasma is used in the Avinit installation to clean the surface and ion assistance when depositing functional coatings.

The strength of the discharge current can virtually be arbitrary and is determined only by the thermal properties of cathode and power supply source parameters and may vary in a wide range by changing the arc current (100–200 A). The range of operating voltages discharge is 40–70 V, at a maximum ion current density.

In ion-plasma cleaning using high-density gas plasma, generated by gas plasma generator, there is no problem of metal particles deposition on the surface, and therefore, the potentials can be changed smoothly for parts starting from zero value.

In this case the complete absence of electrical breakdowns in the contaminated portions of the surface is achieved in comparison with the case where a complete cleaning of the surface is provided by metal ions, and thus the original purity of the treated surface is achieved.

When working with argon, the minimum working voltage on the discharge electrodes at the maximum ion current density corresponds to a pressure of $1 \cdot 10^{-3}$ mm Hg. For nitrogen the optimal pressure is slightly lower and amounts to $2 \cdot 10^{-2}$ mm Hg.

Ion saturation current, on the magnitude of which depends the cleaning process performance, reached high values (~3 A). The processing time in this mode varied from 10 to 15 minutes. This treatment is sufficient for transition to the mode of sputtered metal treatment by ions.

Stage 3 – Ion treatment (cleaning by metal ions).

The main process parameters during the ion treatment is ion current (ion current density), which is used to treat the product surface, the energy of ions bombarding the surface, and the treatment time.

The ion energy is adjusted by changing the accelerating voltage.

The density of ion current on the surface of the treated product is set by the ion current density of ion source that has its own regulation.

Electric arc discharge plasma treatment was started immediately after suspension of treatment in gas plasma.

Smoothly increasing the voltage supplied to the product, the parameters of the arc discharge are brought to the value set by technological process.

The mode of arc sources operation was experimentally chosen so that at continuous mode of the arc combustion the temperature of the products would rise from 573–623 K to 800–823 K while reaching the accelerating potential of 800 V within 8–10 min.

This made it possible to avoid the intensive creation of micro arcs, which can be caused by increased gas emission due to additional heating of the product surface and equipment during treatment, insufficient degree of pre-cleaning of the equipment and products before the chamber vacuum treatment, etc.

The stage of ion treatment can include treatment not only in pure inert gas, but also with impurities of other gases (O_2, N_2 etc.), that is, to carry out plasma chemical treatment.

Table 4.3 shows the results of the experiments performed on the witness specimens in conditions that meet the real conditions of the proposed coating use, in order to study the effect of different stages of pre-treatment of the surface on the adhesion and quality of coatings obtained with the optimal structures provided in the previous Tables 4.2, 4.3 (para 1.5 of Table 4.2, para 2 of Table 4.3). Coating was deposited under optimal process conditions (para 1.5 of Table 4.2).

To improve the adhesion characteristics of the coating in the course of coating, stabilizing annealing was carried out every 50 layers at the same temperature without deposition of coating by turning off nitrogen supply and increasing the displacement potential on the parts to stop coating deposition.

The experiments we performed (Table 4.4) allowed to find the optimal parameters of the stabilizing annealing process (para 2 of Table 4.4), which significantly improves the adhesion characteristics of the coatings obtained.

In the course of coating deposition stabilized annealing is performed every 50 layers at the same temperature without coating deposition by turning off nitrogen supply and increasing the displacement potential on the parts to stop coating deposition.

Carrying out process of ion-plasma purification and formation of layers with predetermined recurrence periods and thicknesses of individual layers during vacuum-arc deposition of the protective coating, is performed by programmatically set cycle schemes that provide program-synchronized control of pressure regulators of inert and reaction gas parameters.

The processes of multi-stage ion-plasma purification, subsequent vacuum-arc deposition of a protective erosion-resistant coating that contains layers based on titanium nitride, and stabilizing annealing of the coating is carried out in a single vacuum chamber within a single process cycle.

Table 4.3 Experiments to study the effect of different stages of surface pre-treatment on adhesion and coating quality

№	1.1. Treatment in glow discharge plasma						1.2 Treatment in high-density argon plasma						1.3. Treatment with metal ions						Common layer characteristics		
	Process parameters			Plasma characteristics			Process parameters			Plasma characteristics			Process parameters			Plasma characteristics					
	Voltage U_c, V	Pressure Ar P_{N2}, Pa	Time, min.	Plasma density n, cm^{-3}	Temp. of electr. T, eB	Ionization degree α, ion/atom	Current I_p (Ti), A	Voltage U_c, V	Pressure Ar P_{N2}, Pa	Plasma density n, cm^{-3}	Temp. of electr. T, eB	Ionization degree α, ion/atom	Current I_p (Ti), A	Voltage U_c, V	Time, min	Ions energy, eB	Current density j, mA/cm^2	Time, min	Adhesion	Total thickness, μm	Microhardness, MPa
Glowing 1.1																					
1.1	800	$1 \cdot 10^{-1}$	15	$1 \cdot 10^{7}$	0.1	10^{-7}	–	–	–	–	–	–	–	–	–	–	–	–	Satisfactory	15	20.000
1.2	1.300	$1 \cdot 10^{-1}$	30	$2 \cdot 10^{7}$	0.15	10^{-7}	–	–	–	–	–	–	–	–	–	–	–	–	Satisfactory	17	19.000
Glowing + ions Ti 1.1 + 1.3																					
1.3	1.300	$1 \cdot 10^{-1}$	30	$2 \cdot 10^{7}$	0.15	10^{-7}	–	–	–	–	–	–	100	90	10	300	0.1	15	Good	15	19.000
1.4	1.300	$1 \cdot 10^{-1}$	30	$2 \cdot 10^{7}$	0.15	10^{-7}	–	–	–	–	–	–	120	100	15	1.000	0.2	30	Good	13	19.000
Glowing + DVDR + ions Ti 1.1 + 1.2 + 1.3																					
1.5	1.300	$1 \cdot 10^{-1}$	30	$2 \cdot 10^{7}$	0.15	10^{-7}	100	100	$1 \cdot 10^{-3}$	$6 \cdot 10^{9}$	5	10^{-4}	120	100	15	1.000	0.2	30	Very good	20	18.000
1.6	1.300	$1 \cdot 10^{-1}$	30	$2 \cdot 10^{7}$	0.15	10^{-7}	120	200	$1 \cdot 10^{-3}$	$6 \cdot 10^{9}$	5	10^{-4}	120	100	15	1.000	0.2	30	Very good	20	17.000
1.7	1.300	$1 \cdot 10^{-1}$	30	$2 \cdot 10^{7}$	0.15	10^{-7}	120	500	$1 \cdot 10^{-3}$	$6 \cdot 10^{9}$	5	10^{-4}	120	100	15	1.000	0.2	30	Very good	25	19.000

Table 4.4 Experiments to select parameters of stabilizing annealing

№	Substrate material	Parameters of stabilizing annealing	Process parameters				Common layer characteristics	
			Nitrogen pressure P_{N2}, Pa	Displacement potential, U_c, V	Time, min.	Temperature	Adhesion	Microhardness, MPa
1	1.4021 Ge	Every 30 layers Ti-TiN	0	120	3	At $T_п$	Good	18.000
2		Every 50 layers Ti-TiN	0	120	5	At $T_п$	Very good	18.000
3		Every 100 layers Ti-TiN	0	120	5	At $T_п$	Very good	18.000
4		Every 150 layers Ti-TiN	0	120	5	At $T_п$	Good	18.000

Thus, as it follows from Tables 4.2, 4.3, coating deposition at optimal process modes (para 1.5 of Table 4.2) and stabilizing annealing (para 2 of Table 4.3) allows to get the best results for adhesion of multilayer protective coatings, as evidenced by the lack of chipping and local sublayers.

The best results for adhesion and quality of coatings are achieved at optimal modes of the process in para 1.5 (Table 4.3).

It is under these conditions that deposition of erosion resistant multilayer coatings for turbine blades was carried out.

Currently we possess unique equipment at our disposal and the technology of depositing coatings developed by us, on the complex configuration and a large length elements, as well as coating depositing on the parts inner surface.

Certain elements of our nanomaterials, nanotechnologies and equipment are unique, and protected by patents while having no analogues.

The coating deposition is carried out in the Avinit installation.

After all shape-generating mechanical treatments before coating deposition, standard operations of thorough degreasing in an ultrasonic bath, rinsing in petrol-acetone solvents, drying in a drying Cabinet at a temperature of 60 °C are performed.

The blade is placed in special technological tools, in a vacuum chamber of Avinit installation where vacuum of at least $2.0 \cdot 10^{-3}$ Pa is created.

Vacuum-plasma deposition of underlying metal is consistently preceded by three stages of treatment that include a product surface treatment in a glow discharge plasma of inert argon gas, the surface treatment in high-density plasma of two-stage vacuum-arc discharge of inert argon gas and, finally, ion-beam treatment with metal ions in accordance with optimal process modes.

Carrying out such a three-stage treatment ensures high quality surface cleaning before coatings are deposited and firmly bonded coatings are obtained. Then a multicomponent coating is formed by vacuum arc deposition from a plasma phase in the environment of the nitrogen reaction gas with ion bombardment under optimal process modes.

Thus, Avinit structures of multilayer coatings were developed, as well as equipment and technologies for their deposition on long (1.500 mm) and large-dimension (100 kg) parts of turbines of thermal and nuclear power plants (working blades of turbines, rails-gear wheels, shafts, etc.).

The developed coatings were deposited on a batch of steam turbines serial blades (1.300 mm long) to protect against corrosion-erosion damage.

For control purposes the witness specimens were installed in different parts of the blades — on the convex and curved surfaces, in the areas of shrouding, feather, and blade locking pieces (Table 4.5).

The most thorough inspection of working blades with developed coatings did not reveal any damage of coatings on all sections of the blades.

Table 4.5 Characteristics of coatings on witness specimens located in different blade areas

<table>
<tr><th rowspan="2">№</th><th rowspan="2" colspan="2">Areas of witness specimens location</th><th colspan="3">Common layer characteristics</th></tr>
<tr><th>Adhesion</th><th>Total thickness, µm</th><th>Microhardness, MPa</th></tr>
<tr><td>1</td><td rowspan="3">On convex surfaces</td><td>($L_1 = 100$ cm)</td><td>Very good</td><td>15</td><td>20.000</td></tr>
<tr><td>2</td><td>($L_2 = 500$ cm)</td><td>Very good</td><td>16</td><td>20.000</td></tr>
<tr><td>3</td><td>($L_3 = 1{,}000$ cm)</td><td>Very good</td><td>17</td><td>19.000</td></tr>
<tr><td>4</td><td rowspan="3">On curved surfaces</td><td>($L_1 = 100$ cm)</td><td>Very good</td><td>15</td><td>20.000</td></tr>
<tr><td>5</td><td>($L_2 = 500$ cm)</td><td>Very good</td><td>16</td><td>20.000</td></tr>
<tr><td>6</td><td>($L_3 = 1{,}000$ cm)</td><td>Very good</td><td>17</td><td>19.000</td></tr>
<tr><td>7</td><td colspan="2">In shrouding area</td><td>Very good</td><td>19</td><td>18.000</td></tr>
<tr><td>8</td><td colspan="2">In feather area</td><td>Very good</td><td>20</td><td>18.000</td></tr>
<tr><td>9</td><td colspan="2">In the lock area</td><td>Very good</td><td>12</td><td>20.000</td></tr>
<tr><td colspan="6">L – distance from blade locking piece</td></tr>
</table>

Coating was deposited under optimal process conditions (para 1.5 of Table 4.2, para 2 of Table 4.3).

According to the metallographic studies of the samples, the low-temperature ($T<200$ °C) modes ensure formation of high quality cohesive coatings after the coating have been deposited. Coating deposition at temperatures not exceeding 150–200 °C does not reduce the output surface hardness. The coatings microhardness has values in the range of 15.000–20.000 MPa (for Ti-N based nanocoatings).

The coatings have good adhesion to the substrate material.

No cases of coating peeling were observed when applying scratch mesh. The nanocomposite coatings had a layered structure of layers of appropriate composition with a thickness of ~10–15 nm.

Coating deposition is a finishing operation — no further refinement of the parts surface is required after manufacture (Fig. 4.2–4.4).

A batch of blades with developed coatings installed as a part of the turbine for operational testing at NPP (Paksi, Hungary).

Fig. 4.2 Turbine blades with developed protective coating using new complex plasma chemical nanotechnologies and equipment (1.300 mm long)

Fig. 4.3 Turbine blades in the course of coating deposition

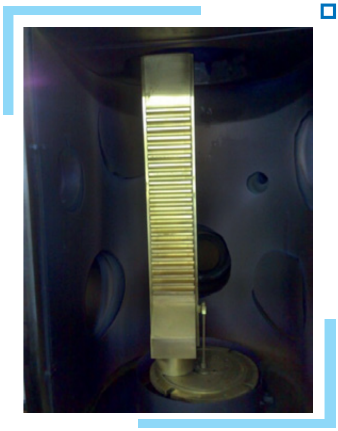


Fig. 4.4 Turbine rail tooth gear with protective coating (length – 1.000 mm, weight – 70 kg)

5

Implementation of Avinit technologies into serial production

Joint work with OJSC "Turboatom" (Kharkiv, Ukraine) is being successfully carried out for development and industrial implementation of innovative wear-resistant anti-friction coating methods based on nanotechnologies in order to enhance the lifetime of various responsible prats of steam and nuclear turbines.

Based on the implemented fundamental and applied researches there have been developed the innovative technologies of focused ion-plasma modifying the materials' surfaces, and deposition of functional multilayer Avinit coatings, using high-density low-temperature strongly disbalanced plasma.

These technologies are implemented in various fields of machinery including protection of steam and nuclear turbine parts, being produced by OJSC "Turboatom" for the needs of thermal and nuclear power plants.

Vacuum-plasma wear-resistant and corrosion-resistant coatings are intended to protect the parts of vapor distribution and regulation systems of steam turbines from corrosion and wear.

There have been found unique solutions to secure reliable functionality of vapor distribution and regulation systems of steam and nuclear turbines that operate in hinged joints and slider bearings, as well as in nodes of sealing the shut-off elements in conditions of dry wind friction and friction in vapor, wet steam, and water environment under the temperature up to 320 °C.

High hardness and exclusively high bonding strength of coatings with various substrates, high corrosion and erosion resistance, inertia under working temperatures, allows for significant enhancement of turbine parts' reliability and lifetime.

There have been developed and agreed the technical specifications for vacuum-plasma deposition of wear-resistant coatings on turbine parts, produced by OJSC "Turboatom". Given technical specifications are applicable to the coatings of the turbine parts operating in the friction nodes with specific pressure on the contacting surfaces up to 200 kg/mm^2, in conditions of dry wind friction and friction in wet steam, and water environment under the temperature up to 580 °C.

Deposition of multilayer antifriction wear-resistant Avinit coatings on working surfaces of friction pairs in slider bears, as well as surface hardening of rods and sleeves of high-pressure control and stop valves, that operate under high temperatures 280–565 °C, ensure a sharp decline in friction (3–4 times). And therefore the lack of abrasions on the friction surfaces allows for the 5–8 times enhancement of turbine vapor distribution system's lifetime when operating under the vapor-air environments.

This allows achieving: durability test by 10–30 %; resistance to drip erosion — 2–3 times; corrosion resistance — 15–30 times.

Developed coatings are introduced into relevant technical normative documentation.

According to technical documentation and technical specifications there have been developed deposition processes of required coatings with high adhesive, corrosion and hardening characteristics, on various complex parts of vapor distribution system (hinges, detents, guides, sleeves, etc.)

These technologies allow achieving certified high-quality coatings (preservation of roughness and mechanical characteristics of coated parts in full compliance with the demands of technical documentation, achievement of required high hardness, coating thickness and composition uniformity).

Developed coatings comply with the international standards and by some parameters are superior to them (Table 5.1). This refers to both coating materials and its production technology. Some elements of developed nanomaterials, nanotechnologies and equipment are unique and have no counterparts.

Works on industrial realization of the developed newest technologies of drawing wearproof antifriction coatings with use of nanotechnologies on details of steam and atomic turbines made by OJSC Turboatom are carried out.

Parts with multilayer anti-friction wear-resistant Avinit coatings have been put into serial production and are successfully operated in turbines manufactured by OJSC Turboatom for Paks NPP (Hungary), Armenian NPP (Armenia), Novocherkassk TPP (Russia), Kazakhstan Aksu TPP and many others (Fig. 5.1, 5.2).

Table 5.1 Avinit multilayer antifriction wear-resistant coatings

Features. The coatings are applied using nanotechnology. The thickness of the coatings is 5–10 μm. Preservation of a class of cleanliness of a surface. Excellent adhesion to the base.	**Application.** Antifriction wear-resistant coatings to improve the tribological characteristics of friction pairs in plain bearings.
Application of multilayer antifriction wear-resistant coatings on the working surfaces of friction pairs in plain bearings allows to reduce friction by 3–4 times, increase by 5–8 times the total service life of turbine steam distribution mechanisms (Fig. 5.3).	**All coatings are guaranteed to be of high quality. Coated parts undergo strict quality control. All products are certified and corresponds to TU.**

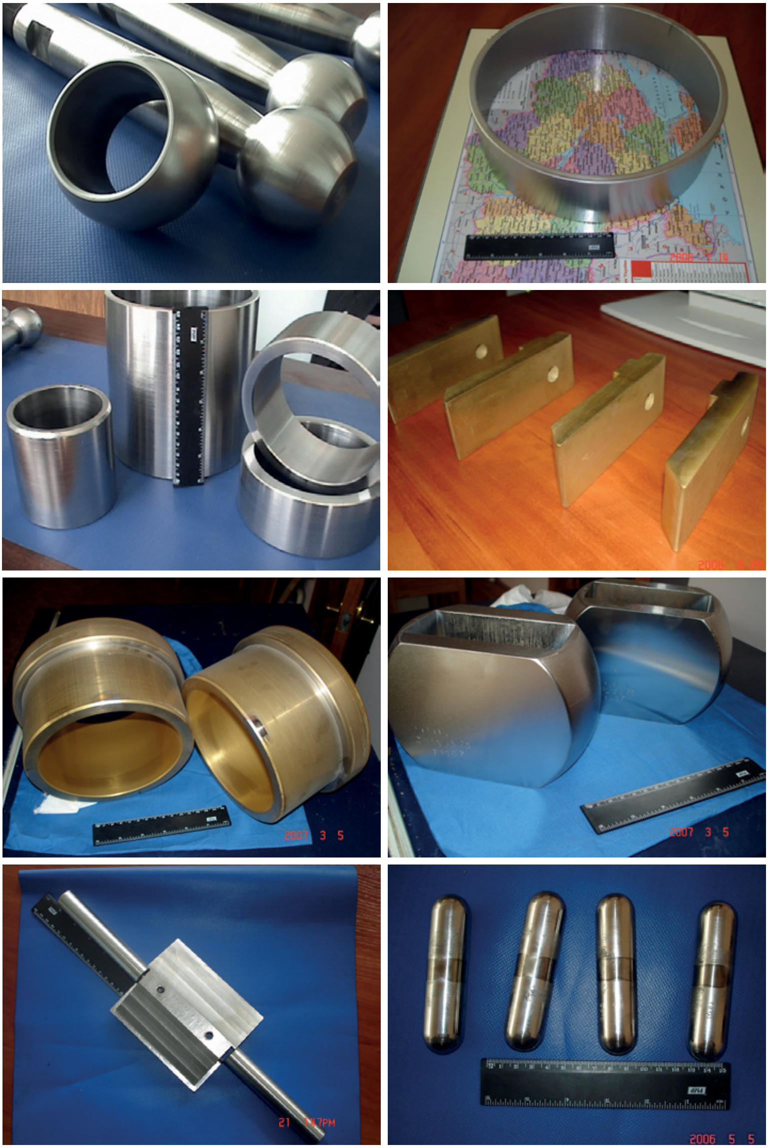


Fig. 5.1 Elements of mechanisms of steam distribution and steam regulation of steam turbines working in hinged connections and sliding bearings with wear-resistant antifriction nanocoatings Avinit

Fig. 5.2 Elements of mechanisms of steam distribution and steam regulation of steam turbines working in hinged connections and sliding bearings with wear-resistant antifriction nanocoatings Avinit

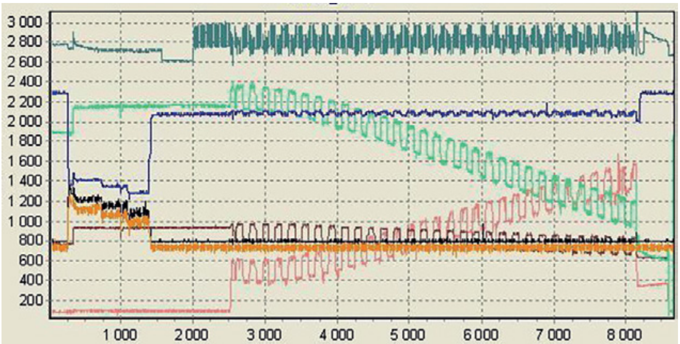


Fig. 5.3 A fragment of the protocol of the automated control system of the technological process of nanocoating

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Abbreviation

GTE	gas turbine engine
GTU	gas turbine unit
CVD	chemical vapor deposition
PVD	physical vapor deposition
MSVI	mass spectrometry of secondary ions
SEM	scanning electron microscopy
SIMS	secondary ion mass spectrometry
EPMA	electron probe X-ray microanalysis
VAC	volt-ampere characteristic