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Hi-tech technologies based on advanced
fundamental research

Improving the performance, reliability and service life of aviation technology products based on the innovative vacuum-plasma nanotechnologies for application of avinit functional coatings and surfaces modification

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 of aerotechnical purposes 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 nonsteady 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 (plasma-chemical CVD, vacuum-plasma PVD (vacuum-arched, magnetron), processes of ionic saturation and ionic surface treatment, 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 of physicochemical, mechanical complex and other properties are considered.

The issues of development of experimental-industrial technologies Avinit and industrial implementation of the developed technological processes to increase of operational characteristics of aerotechnical products are addressed in detail.

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

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

Keywords

CVD and PVD vacuum plasma processes, characteristics of functional coatings Avinit, ion-plasma diffusion surfaces modification, development of nanotechnologies Avinit.

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Preface

The latest trend in the creation of new modern materials with record operational characteristics when working in extreme conditions is associaed with the research and development of nanostructured materials and nanotechnologies.

The monograph offered to the reader presents the results of the authors' research in the development and practical implementation of the latest nanomaterials and nanotechnologies Avinit to improve the performance of materials.

The developed vacuum-plasma nanotechnologies of Avinit are based on the use of gas-phase and plasma-chemical processes of atomic-ion surface modification and the formation of functional nanolayer coatings.

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

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

The monograph presents the results of the authors' research from conducting broad fundamental research to the development of pilot technologies and their serial implementation in the most modern industries — aviation, mechanical engineering, power engineering, instrument making, rocket and space technology.

1

Introduction

Research and production corporation JSC FED has been successfully developing and practically implementing the latest nanomaterials and nanotechnologies of multicomponent coatings (mono- and multilayer, nanostructured, gradient) of various functional purpose (antifrictional, hardening, etc.) for more than 15 years, to enhance the materials operating properties, as well as components and parts of aviation units, and develops technological processes of coating deposition and equipment for their implementation [1—9].

Fig. 1.1 Joint-stock company FED

The latest trend in the creation of new contemporary materials with unprecedented characteristics in roughness, wear resistance, ability to work in extreme conditions is associated with the research and development of nanostructured materials and nanotechnologies.

The transition to the nanoscale allows to form multicomponent compositions with structural elements ranging in size from several hundred to just several nanometer units. Such materials, when compared with materials of the same composition having the usual structure, may have several times higher corresponding characteristics with respect to tribological and other properties.

Multicomponent multilayer solid and superhard coatings exhibit higher wear resistance and tribological characteristics compared to single-layer coatings based on one combination.

Among the coating methods, a special attention is paid to the methods of forming coatings from ionized atomic and molecular fluxes. The ability to vary the energy of ionized particles of the condensed matter flux within a wide range (from one to hundreds and thousands of electron-volts) allows to effectively influence most of the coatings characteristics important in practical terms (density, adhesion, structure, etc.) and thereby achieve the highest values of the corresponding indicators in comparison with other methods and to create multicomponent composite materials in non-equilibrium conditions of their formation.

The main feature is:

- the use of vacuum-plasma processes activated by non-equilibrium low-temperature plasma;
- the transition to the nanoscale for the deposition of multicomponent, including multilayer and nanolayer coatings, due to their wide possibilities to form coatings of different materials in different structural state, including nanostructural, while obtaining nanofilms with predetermined characteristics, maximum density, adhesion and other characteristics of the coating quality.

Methods for the implementation of vacuum plasma deposition, and the appropriate equipment for their implementation are chosen as basic ones.

The technologies are based on the processes of atomic-ionic surface modification and the formation of nanolayer coatings of various elements and chemical compounds under the conditions of non-equilibrium low-temperature plasma effect.

The peculiarity of the developed coating processes lies in their integrity: different coating methods (plasma-chemical CVD [10–15], vacuum-plasma PVD (vacuum-arc [1–9], magnetron [16–18]), processes of ionic saturation and ionic surface treatment) are combined in one technological cycle.

Table 1.1 Hi-tech coating deposition combined techniques

Chemical vapor deposition methods (CVD and PECVD) (gas-phase, plasma-chemical [10–15]). Vacuum-arc coating deposition method [1–9]. Magnetron coating deposition method [16–18]
Ion saturation (nitriding etc.) [35–37]. Precision plasma nitriding in anhydrous environments [35–41]
Ion etching, purification

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

2

Avinit equipment designed for multilayer functional coatings

To deposit functional multilayer composite coatings experimental and technological vacuum-plasma automated cluster Avinit [19] was developed and created, which allows to implement complex coating methods (plasma-chemical CVD, vacuum-plasma PVD (vacuum arc, magnetron processing) ion surface saturation and ion surface treatment) combined in one technological cycle.

Fig. 2.1 Vacuum-plasma cluster Avinit

Technological vacuum-plasma automated cluster Avinit created by modernization of existing industrial equipment of ion-stimulated deposition and diagnostics equipment of nanosized coatings due to introducing new microprocessor power systems, synchronization and control of processes of synthesis and diagnostics of complex control methods into the equipment to achieve purposeful process control.

Technological vacuum-plasma automated Avinit cluster for obtaining multi-component multilayer hard and superhard materials and coatings is a complex high-vacuum unit with numerous power sources of various types (gas-phase and vacuum-arc evaporators).

It consists of technological units and several functional units:

- Avinit C unit for deposition of hard and superhard multilayer and nanolayer functional coatings by the method of modernized vacuum-arc deposition;

- Avinit V unit for gas-phase (CVD) and plasma-chemical (PECVD) coating deposition on parts internal and external surfaces;
- Avinit M unit for magnetron sputter coating;
- Avinit N unit for ion-plasma treatment;
- Avinit E unit for carrying out ion etching and purification processes;
- Avinit T unit for thermal and thermochemical treatment.

2.1 Process units

2.1.1 Sources of energizing the Avinit installation

The Avinit installation includes the power systems required to implement the coating methods provided for in the Avinit installation.

When choosing the method and device type for ionization of all types of evaporators vapors, it is considered appropriate to minimize the expansion of the installation with new power supplies and increase its overall capacity. Such possibilities exist if to take into account that this installation must be equipped with different types of evaporators that can't be operated simultaneously and use unused power sources.

To carry out ionization of vapors, power supplies with voltages from several hundred volts to several kilovolts and with amperage from 1 A up to tens of amperes are required, depending on the mode of discharge excitation.

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

Vapors ionization methods using arc discharge require high current (tens and hundreds of amperes) power supply sources up to 100 V, with which all serial vacuum plasma deposition installations are equipped, provided for in the Avinit installation.

The Avinit installation also includes the use of plasma sources, with which deposition magnetron sources of DC and HF current are equipped.

When using flow charts associated with the use of a glow discharge, the most effective are the methods in which a non-independent glow discharge is used. The presence of DC magnetron evaporator power supplies in Avinit installation enables, to ensure implementation of nonindependent glowing discharge schemes using filament-type cathodes.

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

It is advisable to use a high-voltage ion etching and substrates cleaning rectifier that is the part of the 'Avinit' installation to create the potential difference required for its combustion and feeding drawout potential to the substrate. Therefore, when choosing a method of excitation of a nonindependent glow discharge, a system with HF excitation is used.

Power sources for the HF discharge system use power sources for HF magnetron evaporators that are included in the Avinit installation having foreseen in them the possibility of matching with inductive load.

The existing system of gas injection and inert gas control system included in Avinit cluster is capable to fully provide the necessary environmental pressure conditions for discharge excitation in the vapors ionization of the vaporized substance at virtually any evaporator operating mode, including operation at minimum power.

Due to the chosen chamber design, the configuration of the magnetic field, the elements forming the gas flux, a homogeneous plasma flux is created which propagates to the substrate.

These methods and means of technological parameters control are used as a basic version.

2.1.2 Modified elements of arcing

The Avinit plant is equipped with modified arcing elements of vacuum arc evaporators. Operation in the conditions of vacuum-arc sputtering unit operation of the Avinit installation shows significantly more reliable operation of the plasma sources compared to standard ignition elements

2.1.3 Separation system unit for deposition of coatings on precision surfaces

During the combustion of the vacuum arc discharge, along with the atomic highly ionized flux of particles, part of the cathode material is transferred to the surface of the coating growth in the form of droplets. Their size, flux density, angular distribution depend on the mode of operation of the vacuum-arc sputter source, and the cathode material. The presence of a droplet component, a significant number of macroparticles generated by the cathode spot of the vacuum arc, in the coating structure, is one of the characteristic negative features of vacuum arc coatings.

During the formation of nanoscale coatings, this causes a significant decrease in the characteristics of the coatings obtained and is one of the serious problems for the practical use of methods based on the vacuum arc discharge application, especially when depositing on precision surfaces.

For deposition of high-quality nanolayer coatings on precision surfaces in Avinit installation, plasma sources operating in DC mode, are equipped with devices for separating plasma fluxes.

The used straight-line island-type separator is one of the simple-to-implement, and at the same time quite effective design.

The separator, mounted on the basis of use, focuses coils of a standard vacuum arc deposition source, has a damper and a system of trap rings made of heat-resisting material which provides reliable protection of the anode against the penetration of the arc spot.

The use of such a straight-line separator provides the formation of plasma fluxes purified from the microparticles of the cathode material, which allows the deposit coatings on the surface of $V\,11-13$ class virtually without change in the class of surface cleanliness.

The rate of growth of the coating using a separator at a distance of 160 mm from the its exit was from 0.8 to 1 μm/h on the substrate D=190 mm, and at D=160 mm deviation from the velocity value in the center does not exceed 10 %.

The efficiency of transporting the separated plasma flux depends to a significant extent on the location of the separating device inside the focusing coil and is selected experimentally for a specific part.

Before coating deposition, the separator must be positioned in the working chamber relative to the cathodes and the part to be covered.

2.1.4 Arc suppression unit

When the traditional PVD deposition is used while operating one-component cathodes in pulse or constant modes, the improvement of mechanical and electronic systems of protection against microarcs and modernization of cathode units and control system, which allowed to significantly expand the capabilities of technological equipment and ensure the deposition of Avinit high-quality coatings of high-class cleanliness up to class 12–13 without compromizing the surface cleanliness class.

This is achieved through the use of effective technologies of surface cleaning in the developed technologies, as well as the prevention of surface damage by micro arcs, for which the Avinit installation provides for a three-level (mechanical, electrical and electronic) arcing system, which ensures high quality of surface cleaning from oxides and other pollution without electric breakdowns.

2.1.5 Plasma diagnostics

Technological control of the plasma state parameters of a substance is a complex task because it combines a set of closely related characteristics, the determination of which requires the use of special methods and mathematical processing of the measurement results.

The parameter group used in the Avinit cluster for plasma diagnostics determines most of the characteristics of coatings, regardless of the method they are achieved and regulated with.

First of all, such parameters are the power of the evaporation sources, the heating temperature of the evaporation devices and auxiliary elements of technological equipment, etc. Control and determination of this group of parameters is important for obtaining stable results on the coatings quality.

The applied methods and means of technological parameters control satisfy the basic requirements for installation and are accepted as a basic option.

2.1.6 Automated system cluster Avinit

A multifaceted, integrated Avinit installation provides a mode of operation for using any of the evaporation devices, systems, and control devices included in its composition, with the simultaneous operation of all the same-type evaporators or alternate operation (in any order) of different types of evaporative devices. The solution of the problem of obtaining multifunctional coatings with necessary characteristics is largely associated with ensuring of complete control of process parameters and their support and control in automatic mode. When the design and the nodes of Avinit installation were developed, the possibility of remote control and monitoring their status (position) by means of appropriate actuators and sensors was provided for.

Vacuum-plasma Avinit cluster is focused not only on a specific technological process, but provides the possibility of implementation of various technological processes, in which various coating methods, and methods of substrate pre-treatment can be used.

To implement processes of multi-component nano- and microstructural coatings formation with controlled composition and pre-set characteristics, the unit is equipped with multi-parameter control system for the technological equipment operation.

The formation of nanolayer coatings with the dimensions of the single-level layers from a few nanometres, and stability of their reproduction requires both end-to-end synchronization in management and maintenance at the specified level of all parameters of coating deposition process and their continuous registration, which can't be achieved without an appropriate system of equipment parameters management and control.

A radical restructuring of all the systems of technological equipment management was carried out on the basis of technology of end-to-end synchronization of equipment systems of ion-stimulated deposition and equipment for diagnostics of nanosized coatings due to the introduction of the new microprocessor power systems equipment, synchronization and control of processes of synthesis and diagnostics and development of control methods complex of process parameters in the coating process for targeted process control.

The reconstruction of technological equipment was performed on the basis of computerization of process control, and software products were developed, which allow to apply nano- and microlayer multicomponent coatings on precision surfaces of high-class purity.

The most acceptable from the point of view of possibility of formation of multicomponent nano- and microstructural coatings with controlled composition using the vacuum-plasma and plasma-chemical processes is our developed technology of end-to-end synchronization of sputtering sources, sources of accelerating potential supply to the products covered, system reaction gases injection, and other installation's systems based on a program set, using the computer to control technological process of coatings deposition and the logging of all important parameters of the installation during the whole technological cycle.

To meet the requirements for the control system of the equipment parameters for the deposition of nanoscale coatings, the development of auxiliary control systems for the operation of high-voltage and arc power sources of the installation was provided for, as well as the unit of reaction gas supply to the vacuum chamber, other units.

The installation is provided with a device for automated remote control of all systems and the installation actuators.

A system for collecting, transmitting, registering, accumulating and processing information from all installation systems and mechanisms was developed to create a parameter control system which allows to more precisely adjust the parameters of each of the sputtering sources. The microprocessor part and the computer control system have an exchange protocol and can be synchronized in time, the ability to adjust the spraying sources parameters allows to obtain gradient coatings. Reaction gases injection into the vacuum chamber is controlled by the computer system.

The gas injection system and the sputter sources control system can also be synchronized in time. Microprocessor subsystems include the control of the vacuum pumping system (both manual and automatic modes), the control of the high-voltage bias source (both gradually and smoothly), and the control of the arc-suppression system for the high-voltage bias source (which provides fast <20 msec level of response to the arc discharge, and leaves the substrate intact).

The control system allows the operator to carry out the process both in a step-by-step mode (testing) and in a program (automatic) mode, and in both modes the data registration is continuous.

Taking into account the peculiarity of the coating deposition processes implemented, the data collection and registration system provides:

- operational control of all significant technological parameters of the coating process throughout the entire technological process;

- possibility of visualization and control of the set technological parameters, their deviation from the set technological modes and visualization of this information on the display in a format convenient for the operator;
- data storage in memory with the possibility of visualizing the protocol as the whole process and at a given moment of time at different stages;
- archiving of all performed coating deposition processes;
- wide possibilities for statistical processing of information and protocols presentation in a graphical form;
- possibility of objective certification of the technological process, regardless of the operator, which allows to use the process protocol as a document, confirming compliance of the coating characteristics with the requirements laid down in the flow chart.

The operator can view data both in real time and from the archive.

Similar features are available for central moments and other specific functions that can be built from a set of controlled parameters. It is possible to control using the installation functionality available to the operator (for example, the accuracy of controlling the potential of the substrate about 5 V in the range of 0 – 2000 V, arc current 1 A in the range of 30 – 200 A). The separations of parameters available to the operator are based on the system of access control to OS Linux resources.

Process data collected can be displayed graphically.

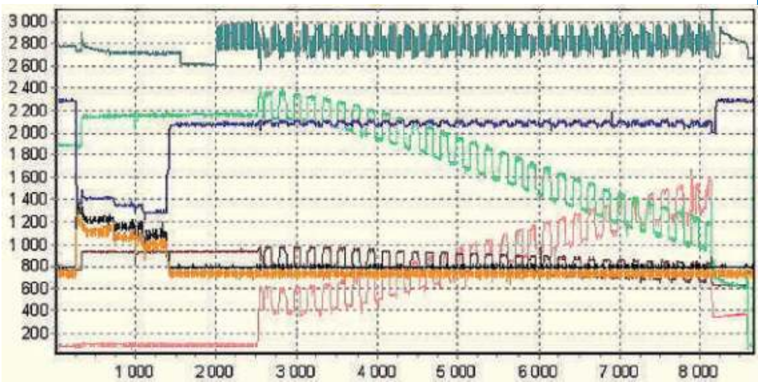


Fig. 2.2 The protocol of the automated system of registration of the basic technological parameters

The use of a special automated system for registration and control of the coating process main technological parameters provides effective parameters control of the technological process at all stages, allows to control the operation of sputtering sources, reaction gases injection and accurate dosing, other installation systems using arbitrary program, which is specified by the operator, as well as logging of all important installation parameters.

The application of the system developed allows to choose the most optimal modes and methods of surface treatment and coating or a combination of them to achieve the maximum technical and economic effect when solving specific problems, to achieve strict compliance with the certified technological modes and to achieve the best indicators of high quality when depositing functional coatings in serial production.

2.2 Functional units

2.2.1 Avinit C unit for deposition of hard and superhard multilayer and nanolayer functional coatings

Avinit C unit is designed for deposition of hard and superhard multilayer and nanolayer functional coatings using the method of modernized vacuum-arc deposition.

The source of the deposited material, when using the vacuum-arc method, is a cathode discharge gap, which excites the arc discharge at reduced pressure (in vacuum).

Fig. 2.3 Avinit C unit

Table 2.1 Technical specifications

Overall dimensions	4.000×2.500×3.000 mm
Weight	not more than 3.000 kg
Temperature of chamber warming-up	80 °C
The number of arc sources	4
Arc current	not more than 150 A
Excitation and support of the arc discharge	automatic
The amount of reaction gases	4
The highest electrical power	not more than 70 KW
Maximal size of the machined parts	400×400×800 mm
Maximal area of the sputtered surface	not more than 94 dm^2
Coating deposition rate	from 1 to 50 μm/h
The number of turning gear spindle rotations	8 rpm
Permissible load on turning gear spindle	not more than 100 kg
Time for the chamber depressurization to 6.5 Pa	not more than 20 min
Time for the chamber depressurization to $6.5{\cdot}10^{-3}$ Pa with high vacuum oil-vapor pump	not more than 30 min

In contrast to the arc discharge at normal (atmospheric) pressure, the vacuum arc discharge occurs in metal vapors, with the discharge is localized in small regions of micron size and moving chaotically along the cathode surface. The energy density in such areas, called cathode spots, reaches 109 W/cm^2.

Due to this within a time of ~5 — 40 nsec. (the resting time of the cathode spot in its chaotic motion), the metal vapor pressure reaches ~10^5 Pa, and the ionization degree of the metal vapor can be close to 100 %. The ion energy in the arc discharge plasma equals to 5 — 20 eV.

These features of the vacuum arc discharge, basically, determine the possibilities of the considered method for obtaining coatings with a high degree of adhesion, density, different structural state and phase composition.

The disadvantages of this method include: the isotropic nature of the deposited material spillage which dramatically reduces the utilization rate of consumable material; inability to control the phase composition of the evaporation products; difficulties in controling the energy of condensed particles when depositing coating on dielectric surfaces which impairs the optimization of the film deposition process, the relatively high initial cost of the equipment used and its relative sophistication.

The disadvantages of the above methods are absent in the vacuum-arc deposition method using plasma vacuum-arc accelerators. By using an arc discharge in the material vapor of one of the electrodes only as a generator of highly ionized plasma, it is easy to control several process characteristics. For example, by changing the negative potential of the substrate holder, it is possible to adjust the energy of the deposited particles and to control the coating adhesive characteristics.

Vacuum arc methods have advantages in providing the highest adhesion values, the ionization degree in the plasma flux can reach 70—90 % which makes it possible to effectively influence most of the coating characteristics due to the controlled change of the substrate potential.

One of the disadvantages of this method is the presence of a dropping component in the atomic flux directed to the substrate.

Plasma vacuum arc accelerators have a high material utilization ratio close to 100 %, and allow to apply both metal and dielectric coatings, films of refractory and fusible metals, alloys, and semiconductors.

Since the process takes place in high vacuum, the contamination of the condensate films by the working gas is excluded, and the residual gases are effectively absorbed on a special film during the first period of operation.

Due to the almost complete absence of harmful wastes in the deposition of vacuum coatings, the coating does not cause any environmental problems related to the need for their disposal or destruction, which causes significant advantages of vacuum technologies in comparison, for example, with electrochemical methods, as well as with methods of chemical-thermal treatment.

The vacuum-arc method allows:

- obtain a coating of virtually any metal, alloy or compound;
- while conducting deposition processes in reactive gaseous media (N_2, O_2, CH_4, etc.), obtain coatings of oxides, nitride, carbides of metals, oxycarbonitrides and more complex compounds;
- while applying a negative potential to the product, conduct pre-ion-plasma surface cleaning, heat the product and maintain its temperature at the required level, modify the structure of the growing coating and change its other characteristics.

The most effective application for:

- Application of thin film reinforcing, wear- and erosion-resistant coatings on cutting tools, press molds, machines and mechanisms parts, etc.;
- deposition of protective and protective-decorative coatings in chemical, machine-building industries, automotive industry, production of medical tools, consumer goods, etc.;
- coating for corrosion protection in aggressive and especially corrosive liquid and gas environments;
- obtaining coatings and materials.

2.2.2 Avinit V unit for gas-phase (CVD) and plasma-chemical (PECVD) coating methods on parts internal and external surfaces

The coatings formation by CVD method occurs due to the flow of products of heterogeneous decomposition (hydrogen reduction) of metal-containing chemical compounds on the heated surface that are in the reaction volume in a gaseous state.

Fig. 2.4 Avinit V Unit (CVD)

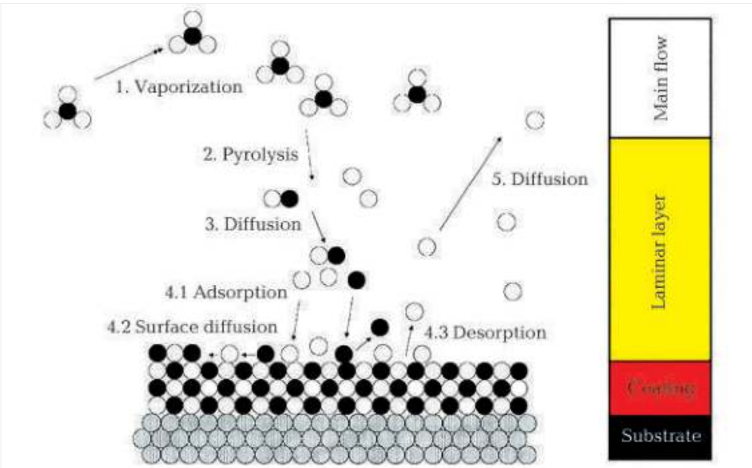


Fig. 2.5 CVD flow chart

Due to the high mobility and intensity of the mass transfer processes inherent in gaseous media, the CVD coating method has an exceptional 'covering' ability.

The ability to provide high mass fluxes of metal-containing compounds in the gaseous state to the coating surface, allows to implement coating processes' high productivity in which the growth rate can reach from several hundred microns to several millimeters per hour.

The high surface mobility of adsorbed metal-containing compounds allows to obtain coatings in CVD processes with a density close to theoretical, at temperatures of ~0.15 – 0.3 from the melting point of the material, which is not available for other coating methods, and to form very high quality epitaxial coatings.

The relative ease of cleaning from most impurity elements at the stage of obtaining metal-containing compounds due to the selectivity of the processes of chemical interaction of the initial products on a heated surface, additional distillation in the process of its transition into a gaseous state from the solid or liquid state, in which these compounds are usually found under normal conditions, cause a high degree of purity of coatings obtained by the CVD method.

Plasma support (PECVD) is a powerful tool for influencing both the kinetics of CVD coating processes and coating properties. The use of different methods of plasma excitation in the reaction volume and control of its parameters allows to intensify the coatings growth processes, to shift them to a region of lower temperatures, and makes the processes of coating of a given microrelief and structure formation, impurity composition and other characteristics of the coating, more controlled.

The methods advantages:

- Unique structure and properties of ion-condensed materials (amorphous, nanocrystalline, microlayer structures, ultra-high hardness, high purity, exceptionally high adhesion strength with various substrates, special physicochemical, electrophysical and other properties).
- Ecological purity and a wide range of coatings (from virtually any element), including W, Re, Ta, Nb, Cr, V, Ti, Al, B, their alloys, refractory oxides, carbides, nitrides, and metal-ceramic compositions based on refractory metals and oxides.
- The possibility of varying the coating deposition speed within a wide range from several units to several thousand micrometers per hour, which allows to obtain in a controlled manner both thin films with a pre-set structure and composition, and to form products with wall thickness up to 10 and more mm from different (including difficult-to-treat, e. g., W) materials and unique non-mixed alloys (e. g., Mo-Cu) (ion-plasma metallurgy).
- The highest 'coating' ability among all known methods, providing the formation of homogeneous thickness coatings on the surfaces of complex geometry, including blind holes, internal cavities and pipes with $L/d \gg 1$ (L – pipe length, d is its diameter).

Among the known high quality coating methods, the CVD and PECVD methods are out of competition in most cases where it is necessary to:

- Deposit uniform, thick, high-density coatings to products of complex shape with a developed surface.
- Obtain a coating of refractory, hard-to-process metals, alloys and compounds with a density close to theoretical and high purity, to form from them self-supporting products of different geometries.
- Deposit coatings on powders and other bulk materials, impregnate (seal) porous structures.

The most effective is the use of CVD and PECVD methods for:

- Deposition of heat-resistant and thermal-resistant coatings of refractory metals, alloys, compounds on units and parts of machines, devices operating in conditions of high thermal loads.
- Deposition of coatings to ensure protection against corrosion in aggressive and especially aggressive liquid and gas environments, to protect against high-temperature and atmospheric corrosion.
- Production of crucibles and technological equipment for production of especially pure semiconductor materials, materials for optical and optoelectronic devices.
- Obtaining semiconductor materials and epitaxial coatings.
- Obtaining conductive, barrier and other functional coatings for the products of the radioelectronic and microelectronic industries.
- Metallization of bulk materials (diamonds, powders), impregnation of graphite, fibrous materials.

Features of Avinit V unit for CVD methods implementation. The Avinit V unit is designed for the implementation of coating deposition processes by thermal decomposition of organometallic compounds, mainly hexacarbonyls Mo, W, Cr and their compounds with nitrogen, carbon, and the like.

The critical temperatures of the hexacarbonyls of these metals are virtually the same and lie in a technologically convenient working area, whereby multilayered, multicomponent structures can be grown within a single growth cycle. Certain specificity related to the physical properties of the organometallic compounds used imposes its requirements for the equipment used.

The schematic diagram of the gas-phase unit of the Avinit V installation is shown in Fig. 2.6.

Some specificity related to the physical properties of the organometallic compounds used, imposes its requirements for the equipment used.

The following elements are located immediately in the working chamber:

- Guide channel with a nozzle — for direct supply of reagents to the surface treated.
- Induction heating — the supplied power to the HF source is 1 kW.
- Potential supplied to the sample — high voltage ±1.5 kV.
- Reflectors, pre-swirl nozzles — the elements of the gas-dynamic system.

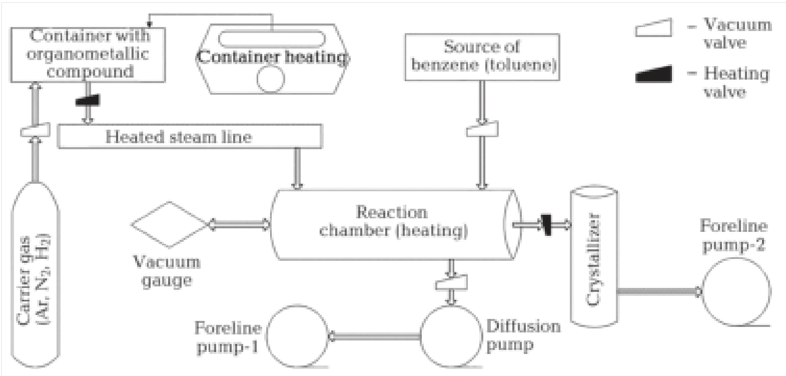


Fig. 2.6 Schematic diagram of the Avinit V gas-phase unit

Organo-metallic reagents are placed in a special heated container with a heated valve at the outlet.

The container temperature can reach 80 °C. An auxiliary carrier gas, argon Ar, nitrogen N_2 or hydrogen H_2, may further pass through the container. Then, through the heated steam line (t=50 °C), the gas mixture is fed into the working chamber. The walls of the chamber are also heated to 50 °C.

Pumping out of the working chamber is carried out in two directions in parallel. On the one hand, pumping is carried out by a diffusion pump — to ensure maximal de-gassing of the working chamber before the technnological process.

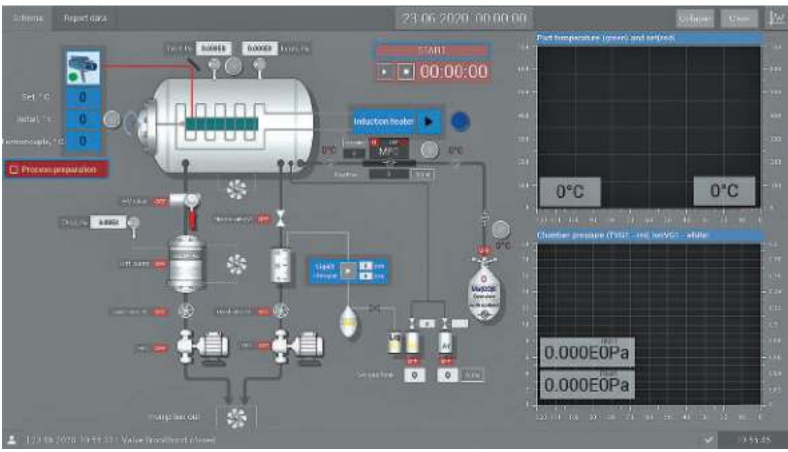


Fig. 2.7 Avinit V gas-phase mnemonic diagram

Pumping is carried out by a forevacuum pump during the technological process itself. For the recovery of expensive working reagents, a crystallizer is installed in before the fore vacuum pump, on which unreacted material is condensed. Due to the fact that the sublimation temperature of the metal-containing compounds used lies within the range of room temperatures, recrystallization processes become especially significant.

Therefore, all components of the equipment are made warmed up.

To heat the products coated in the Avinit V unit, a HF inductor with a working frequency of 3 MHz and an effective power of ~20 kW is used. The sample temperature is controlled by an IR pyrometer through a special window in the chamber door.

The pressure was adjusted by dynamically changing the depressurization rate of the vacuum system within 2.6...13 Pa.

Software package was developed on the basis of the Simple SCADA program for automatic adjustment, registration and archiving of CVD process parameters.

In order to stabilize the technological parameters while developing CVD coating technology for aircraft components, a model version of the control automatic system for sample temperature based on the Arduino Mega 256 microcontroller was developed, which provides:

- keeping the temperature of the working sample during the coating process at a predetermined level, with temperature fluctuation +/–1 %;
- display of the coating process parameters graphs (sample temperature, temperature setting, etc.) on the computer in real time;
- archiving of process data.

A unit of programs for automatic pouring of liquid nitrogen into a nitrogen trap from a shell program in Simple SCADA in a remote mode is developed.

The Avinit V unit is equipped with a device for carbonyl Ostwald ripening from a nitrogen trap, which allows to increase the final carbonyl utilization factor.

Coating deposition on complex-geometry parts involves a number of difficulties.

Typically, the coating is applied on a flat open surface, with any defects or structural features of the surface (grooves, boring through, corners, notches, protrusions) lead to uneven deposition, and sometimes even to coating defects.

A particular problem is the coating deposition on the inner surfaces — holes, tubes, etc., mainly due to the uneven coating along the length of the hole.

In case of the ratio $L/d>1$, such irregularities can be disastrous (resulting in the complete absence of coating at the outlet of the hole).

Gas-phase deposition methods are very promising for coating deposition on the inner surfaces.

Gas-phase coating technologies using organometallic compounds meet current production requirements for the properties of the obtained coatings, as well as their quality and versatility.

From the point of view of practical application, development of processes for obtaining gas-phase functional coatings on complex-geometry precision surfaces of high-grade purity finishing (above class 10) and evaluation of the possibility of using such coatings as candidate materials for friction couples in precision aircraft assembly units, are of particular interest.

To ensure the uniformity of the protective coatings in thickness and properties throughout the entire coated surface, various designs of induction blocks and thermostatic nozzles were developed and tested.

As a result of the research and optimization of the conditions of gaseous metal-containing compounds supply to the products being coated, the geometry and dimensions of induction blocks and gas-distributing devices are determined, which allow to perform a uniform heating and to create uniform flows of metal-containing compounds along the surfaces of the products being coated.

The use of the developed designs of gas-distributing devices creates the pressure difference between the internal volume of the nozzle and the reaction volume in the process of coating, which leads to the formation of laminar flow of vapors of metal-containing compounds directed from the evaporator opening normally to the surface of the product to be coated.

This increases the coating deposition rate due to intensifying the mass transfer processes.

The use of developed induction blocks and gas distributors and planetary movement of products (from 3 to 6 pcs.) allows to obtain uniform thickness and coating composition over the entire surface of the products due to providing the same deposition conditions (temperature, carbonyl vapor concentration, velocity and laminar flow) along the entire the surface to be coated.

In order to stabilize technological parameters while developing CVD technologies for coating aircraft components, an option for the system of automatic control of process parameters has been developed and implemented.

2.2.3 Avinit M unit for magnetron sputter coating

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

Magnetrons (DC and RF) are mounted on the side flange of the installation vacuum chamber.

The diameter of the magnetron target is 120 mm, the maximum intensity of the radial component of the magnetic field at the target surface is 500 E.

The unit is fully integrated with the Avinit N Unit and the Avinit C Unit, enabling the development of hybrid technologies.

Magnetron is supplied from a DC power supply with a strong negative voltage-current characteristic with a power of 2 kW and an idle voltage of 6 kW.

The RF power is fed to the induction block via a matching device from a UV-1 serial generator with an operating frequency of 13.56 MHz and a power of 1 kW.

To excite the RFI-discharge in the gap between the substrate holder and the target, symmetrically to the target axis, a two-turn induction block was installed in the region of a weakly inhomogeneous magnetic field of a magnetron with intensity of 15–20 E.

The induction block is made of a copper tube with a diameter of 4 mm, and the diameter of turns — 180 mm.

The induction block turns are protected by ceramic insulators to prevent the intense sputtering of the surface due to the plasma exciting by due to the high potential of self-displacement and contamination with the sputter coating products. The distance between the target and the substrate holder is 150 mm, though, generally, it may vary.

Fig. 2.8 Avinit M unit

The substrate holder is electrically isolated from the vacuum chamber to be able to supply and adjust the displacement potential of the substrate and to control the total current flowing through it.

Basic features:

- dimensions of the working chamber — ∅ 700×550 mm^2;
- threshold vacuum in the working chamber — 6.6·10^{-5} Pa;
- operating pressure — (0.13–2) Pa;
- 'TORUS 3' magnetron sputtering source by Kurt J. Lesker (USA);
- maximum power — 600 W;
- frequency — 13.56 MHz;
- target diameter — 76 mm;
- supply of gas to the inert and reaction gas chamber — 2 independent lines with regulators — flowmeters from 0 to 200 cm^3/min made by 'Bronkhorst' (Holland);

- measurement of coating rate and coating thickness – Inficon FTC-2800 multi-channel quartz controller by Kurt J. Lesker (USA);
- coating deposition rate – up to 10 μm/h (depends on the power supplied to the target sputtered, the target material, the distance to the substrate);
- dimensions of coated products – up to ∅ 150×100 mm^2;
- the maximum electrical power consumed by the installation, not more than 7 kW.

The Avinit M unit provides coating deposition of metals and their various compounds, including non-conductive, as well as non-metals (carbon, fluoroplast, etc.).

This allows to use the installation to obtain coatings of various functional purposes (protective, hardening, wear-resistant, optical, etc.) and those differing in composition and structure (monolayer, multilayer, including nanolayer and nanostructural).

Such diversity of coatings makes the RF magnetron sputtering method of targets and technologies based on its use, one of the most demanded in a number of areas of contemporary production, as well as in the development of new types of coatings and technologies for their production.

The Avinit M unit for HF magnetron coating is equipped with a system of electronic flow meters for argon and nitrogen to accurately set determine and control the flow and composition of gas mixtures in the chamber during technological processes, as well as to monitor the efficiency of the chamber vacuum pumping.

Additionally, the Avinit M unit is equipped with a voltage stabilizer to protect the power supply circuits of electronic equipment and appliances from damage caused by voltage fluctuations in the shop power supply system.

The Avinit M Unit has an in-chamber rotary actuator (without inserting a rotation shaft into the chamber through a vacuum sealing system) to ensure the ability to deposit coatings (with appropriate equipment) on the outer surfaces of cylindrical, spherical and other shapes, as well as on the inner surfaces of products having the shape of semi-ring, semi-cylinder, hemisphere.

2.2.4 Avinit N unit for ion-plasma surface modification

The Avinit N unit is intended for carrying out processes of ion-plasma surface modification, in particular, for ion-plasma surface treatment, plasma diffusion saturation (nitriding, nitro-cementation, etc.) of parts from steels and alloys in high-density, low-temperature non-equilibrium plasma, precision nitriding in anhydrous environment, processes of diffusion saturation in plasma with hollow cathode.

The Avinit N unit is designed in such a way that it can attach devices for simultaneous magnetron sputtering processes.

Fig. 2.9 Avinit N unit

Basic features:

- dimensions of the working chamber — ∅ 700×700×1.000 mm;
- threshold pressure in the working chamber, PA not more than $1.3{\cdot}10^{-3}$;
- the working pressure in the chamber within, Pa — from $1.3{\cdot}10^{-1}$ to $6.5{\cdot}10^{-1}$;
- supply of gas to the inert and reaction gas chamber — 2 independent lines with regulators — flow meters from 0 to 200 cm^3/min made by 'Bronkhorst' (Holland);
- the number of plasma vacuum arc sources — 4;
- arc current A, not exceeding 150;
- disruption and maintenance of arc discharge — automatic;
- the ion bombardment rectifier has the same parameters as in the Avinit C unit;
- maximum dimensions of the machined workpiece, mm, diameter — 400;
- the number of turning gear spindle rotations, rpm — 8;
- load allowed on the turning gear spindle, kg — not more than 50;
- the greatest electrical power consumption not more than 50 kW.

Avinit N unit includes a gas plasma generator for carrying out the processes of vacuum-plasma high-precision nitriding of steels and alloys in high-density, low-temperature nonequilibrium plasma.

Avinit N unit ensures processes of nitriding the surface of parts made of steels and alloys in high-density nonequilibrium plasma of nonindependent glow discharge, the so-called double vacuum-arc discharge (TSVAD) supported by the burning arc discharge.

Plasma burns evenly in large volumes, providing uniform heating of the parts to the required temperature and nitriding of complex-geometry products of various shapes and sizes, including through and blind holes. Its density is several orders of magnitude higher than the one during ion nitriding in a conventional glow discharge, resulting in 2–5 times more intensive process of the nitrided layer formation in comparison with the traditional processing method of ion nitriding in a glow discharge, and 5–10 times higher in comparison with gas nitriding. This ensures the absence of deformation (buckling) of parts while maintaining the original geometric dimensions after nitriding with a precision of 1–2 μm, totally absent fragile surface layer, typical of traditional nitriding methods.

This allows to avoid finishing grinding of parts and to carry out the operation of precision nitriding 'to the size'.

2.2.5 Avinit E unit for ion etching and purification processes

In the process of depositing functional reinforcing and protective coatings, a very thorough ionic treatment (cleaning) of the products surface is provided for:

1. Ion surface treatment of the product using a glow discharge.
2. Ion surface treatment of the product using an Avinit installation gas plasma generator.
3. Ion surface treatment of the product using metal ions.

The Avinit E unit is equipped with devices to ensure implementation of high-density glow discharge plasma excited in a hollow cathode.

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

Basic features:

- dimensions of the working chamber — ∅ 700×550 mm^2;
- threshold vacuum in the working chamber — $6.6 \cdot 10^{-5}$ Pa;
- operating pressure — (1.3–133) Pa;
- high-voltage plasma excitation source — 2 kV, 2.1 kW;
- dimensions of the hollow cathode — 150×150×150 mm;
- supply of gas to the inert and reaction gas chamber — 2 independent lines with fine regulation inlet valve;
- plasma density control — a universal device for plasma probe diagnostics — 'PlasmaMeter' (Ukraine);
- the maximum electrical power consumed by the installation — not more than 6.5 kW.

The installation provides the possibility of surface treatment of materials in the high density glow discharge plasma in gaseous media of various composition (argon, nitrogen, carbon-containing gases and mixtures of these gases), including internal cavities and channels of specific sizes. The use of high-density glow discharge plasma in the processes of ionic nitriding, and nitro-cementation, allows to reduce, by a several-fold factor, the formation time of hardened layers up to 0.3–0.4 mm in thickness in comparison with gas furnace technologies, while maintaining practically unchanged dimensions of the products treated and preventing the formation of fragile phases on their surface. Therefore, the development of new technologies for surface treatment in high-density plasma is one of the topical areas of advanced technologies perfection in a number of industries.

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

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

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

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

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

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

- purification from retained surface atoms of contaminants;
- heating the substrate material;
- degassing;
- selective etching, which changes the morphology (purity of treatment) of the surface;
- activation of surface atoms, which ensures the flow of plasma-chemical processes of interaction with condensed matter atoms.

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

When using electric-arc methods, pre-ion purification can be carried out by sputtered metal ions while adding to a product an accelerating potential of such magnitude at which the metal self-sputtering coefficient is greater than one (for titanium this potential is close to 1.000 V). If the potential applied is smaller in size, the surface of the product will not be cleaned as metal will condense on it.

It is possible to treat the Avinit installation surface with ions of inert or reaction gas (Ar, N_2, etc.) during the glow discharge ignition.

Due to the lower density of ions in the glow discharge plasma, the intensity of heating and the associated gas release from the surface to be coated will be lower and the intensive formation of microarc discharges can be avoided.

Devices with arc evaporators, in addition to the known advantages, have a number of serious disadvantages. First of all, this concerns the problems arising from ion cleaning during electric arc spraying due to the occurrence of erosion traces from cathode spots of arc discharge (microarcs) which leads to a significant deterioration in the cleanliness of the surface treated, as well as a significant number of macroparticles ('droplet component') generated by the vacuum arc cathode spot.

There are technological problems for which the presence of defects in the coating caused by the metal microparticles is absolutely unacceptable (deposition of functional coatings on precision surfaces, anticorrosive, decorative, optical and other coatings).

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

Effective ion purification is possible if a highly ionized gas plasma is available in the working volume.

In the Avinit installation, taking into account the complexity of methods (plasma-chemical, vacuum-arc, magnetron), as well as the peculiarities of the geometry and precision of the surface of the products to be coated, a gas plasma generator based on a two-stage vacuum arc discharge (TSVAD) was used to replace the metal-gas plasma being used as specialized powerful plasma source of gas plasma for the purposes of high-efficiency ion treatment with cathodic sputtering of the surface with working gas ions, which provides a strong adhesion of the coating to the substrate material and the deposition of high-quality functional coatings.

While carrying out ion-plasma purification using the TSVAD in the presence of gas plasma there is no problem of deposition metal particles on the surface, and therefore, the potentials for the product can be changed smoothly, starting from zero.

Under these conditions, it is possible to achieve a complete absence of electrical breakdowns on the 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, to achieve the preservation of the original purity of the treated surface.

In addition, purification by gaseous rather than metal ions may have the advantage in that the sputtering coefficient of the gas ions is often higher than the sputtering coefficient of the metal ions, and therefore, the process of ionic purification at the same ionic current and ion energy occurs more intensively. And although, the cost of electrical power in the TSVAD installation is twice higher than the cost of a magnetron plant that makes the use of magnetrons for cleaning purposes more widespread, the ability to efficiently clean complex geometry surface products with high purity surface treatment (such as hemispheres) using TSVAD when sputtered targets can be of almost any shape, and the use of magnetrons is ineffective, the use of two-stage arc discharge (TSVAD) is very expedient.

2.2.6 Avinit T unit for thermal and thermochemical treatment and diffusion welding processes

The advantages of the thermal equipment manufactured by our company are in that their designs differ in a number of basic features:

1. For vacuum system:

- high performance pumping system;
- the use of vacuum pumps with high pumping performance and less power consumption, which reduces the time to create a working vacuum and facilitates its maintenance throughout the technological process of heat treatment;
- the use of remote-controlled valves allows automation of the plant's control process to a higher extent and to prevent emergencies arising from operator errors or power outages.

Fig. 2.10 Avinit T Unit to carry out thermal and chemical treatment processes. Own production

2. For the system of parameters support, adjustment and control:

- advanced vacuum measurement system;
- application of advanced vacuum sensors, which have a large resource of trouble-free operation;
- enhanced thermal insulation of the heating unit;
- low-voltage three-phase heater;
- production of new three-phase systems for smooth regulation of heaters power, using the method of phase regulation, on the basis of advanced semiconductor devices;

- replacement of metal heaters and thermal unit lining for heaters and lining of carbon-carbon composite material or graphite, which are more resistant to thermal shocks and have a much greater service life, and also allow to raise the operating temperature in the furnace;
- replacement of single-phase or two-phase heaters for three-phase, which can significantly reduce the load distortion in the shop grid;
- smooth adjustment of the heater power.

3. For the thermoregulation system:

- the use of advanced instrumentation systems — temperature control, which allows to program the entire technological process of heat treatment (the temperature rise rate, the necessary 'shelves', the dwell time on the 'shelves', cooling time, etc.).

4. For the system of gas supply and gas regulation, and providing the necessary gas flows:

- flow and temperature sensors are installed in the cooling system of the furnace to provide control over the furnace cooling, which allows to timely respond to cooling system failures.

Changing the lining design of the working area with the use of advanced insulation materials to reduce heat losses, can significantly reduce the electrical power consumption when heating the melting charge.

5. Furnace control system based on SIEMENS industrial controller:

- installation control system is manufactured using the advanced components, including control systems based on the industrial serial controller from SIEMENS.

The installation control system based on an industrial controller (abandoning of relay circuits) can significantly improve the reliability of the control system, as well as simplify troubleshooting during the installation operation.

The system contains the required number of digital and analog inputs/outputs to control the installation and the process itself.

An industrial operator panel with a display can be used as a visualization tool.

The status of all units and devices during operation, as well as the values of process variables can be visualized on the display graphically in dynamic mode.

The installation control system is designed in such a way that the operator's errors or incorrect actions are excluded to the maximum;

- equipping the furnace with a system of real-time display, logging and archiving of process parameters (temperature, vacuum values), by means of which, curves of temperature, pressure and significant parameters in the working area of the installation are displayed in real time.

The data looks like a graph, referenced to the time of measurement.

At the same time, the temperature and pressure values, with reference to the time of measurement, are archived on the computer hard disk automatically.

At any time, graphs are available for viewing and printing out.

2.2.7 Avinit S unit to carry out electron-beam coating processes

The source of energy that converts the sputtered substance into a vapor state is a focused electron beam with electron energy $1–5 \cdot 10^{-15}$ J. The electron beam is formed by the electron gun and is directed to the surface of the material, evaporates while heating it usually to melting. Due to this, high rates of evaporation of various materials can be reached, including refractory metals, oxides, etc. connections.

Evaporation of materials which are deposited as coating can be done from both non-cooled and cooled crucibles.

The use of cooled crucibles, a high degree of vacuum in the vacuum chamber allows to obtain coatings from high-purity materials.

The modified method of electron beam physical vapor deposition with plasma support due to the ionization of the vapor of the evaporating substance, due to disruption of the discharge between the evaporator and the walls of the vacuum chamber, provides coatings with a greater degree of controllability of properties compared to conventional electron-beam method, while ensuring better adhesion, density, structure and other characteristics.

Technological capabilities. The electron-beam method allows:

- obtaining thin and thick coatings of metals, alloys, compounds, semiconductor and dielectric materials with high productivity;
- obtaining thin-walled products made of refractory and difficult-to-process metals and compounds;
- obtaining coating in highly non-equilibrium metastable states with special properties.

Areas of application:

- deposition of thin and thick-film coatings of various purpose (hardening, wear-resistant, erosion-resistant, anti-corrosion, heat-protective, etc.) in the production of equipment, tools and materials for machine-building, chemical industries, energy-generation sector, etc.;
- deposition of thin film coatings (illuminating, reflecting, protective, interference, filtering, mirror, etc.) on parts of optical systems and devices;
- deposition of hardening, protective and protective-decorative coatings on metals, dielectric materials, glass, plastics in the production of products for various purposes, including consumer goods;
- manufacturing of products made of refractory and difficult-to-process materials intended for operation in extreme conditions.

3

Experimental research and technological developments of the Avinit processes

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

Fundamentally new (PVD and hybrid PVD+CVD) processes of controlled formation of multicomponent nano- and micro-layer coatings in metal-nitrogen and metal-carbon systems using vacuum-plasma (PVD) and plasma-chemical (CVD) methods have been developed:

a) hard and super hard coating:

- *on nitride base* in 'metal-nitrogen' systems [1–9];
- *on carbide base* in metal-carbon systems [11–15];

b) metal-to-metal coatings [10];

c) antifriction coatings based on molybdenum disulfide [20] with impurities of molybdenum, titanium, teflon, copper, etc., obtained by magnetron and CVD methods.

Fig. 3.1 Discussion of scientific issues

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

Multicomponent multilayer nanolayer coatings with high wear resistance and tribological characteristics have been developed [42–45].

The combination of high hardness, durability, and low friction values in Avinit hard and superhard coatings, which are multilayer multicomponent structures, is important when used in friction couples to prevent adhesion.

Their properties are determined not only by the ratio of the elements in the coating and the phases that make up their composition, but also by their structural characteristics.

Coating deposition preserves the surface purity grade (initial roughness corresponds to the 12–13 purity grade).

Low temperature processes ensure deposition of coatings that have good adhesion to the substrate at temperatures not exceeding 200 °C, which does not cause the reduction in substrate hardness.

The main focus of our work on the creation of new functional coatings structures is related, above all, to the needs of aviation units building and is aimed at the development of new materials and industrial technologies for the deposition of multilayer and nanolayer ion-plasma and plasma-chemical coatings and their introduction into the serial production of new aviation products to gradually increase the products service lives and improve their reliability through the use of the developed nanotechnologies.

The properties of the coatings obtained depend on many parameters and a considerable amount of research is required in each case to determine the optimum parameter.

We have studied the influence of the basic parameters on the changing of the obtained coatings properties based on molybdenum, titanium, aluminum, zirconium and their compounds in the form of nitrides, carbides, and oxides.

An important parameter is the temperature of the coating formation.

In many cases, the coating should preserve the mechanical properties of the substrate material, which can be achieved by appropriate heat treatment modes, at which the tempering temperatures do not exceed 180–240 °C.

This also imposes relevant restrictions on the coating temperature of such materials.

Achieving sufficient adhesion of coatings at such temperatures, even for vacuum-arc methods, which in this respect are one of the best, compared to other methods, is not always an easy task and requires careful preparation and selection of modes of vacuum-plasma surface treatment and subsequent coating deposition.

This point was selected as one of the starting points when elaborating the coating deposition modes.

The results of performed studies indicate that when applying coatings by vacuum-arc method in different modes that differ in the time of previous ion-plasma cleaning and the amount of negative displacement supplied to the sample during the coating process, the degree of uniformity of the coating distribution is very sensitive to the parameters of the coating deposition process.

By optimally choosing the process parameters, it is possible to form coatings on sharp edges and on a spherical surface.

However, the sensitivity of the coating deposition to the process conditions uniformity makes it advisable to optimize the process at the stage of the coating process elaboration with respect to model and actual products.

The performed studies are based on the choice of temperature-time parameters of obtaining hardening coatings to improve the wear resistance of the working surfaces of precision friction couples that provide coatings of a given composition with a thickness of 1–3 microns.

3.1.1 Avinit C coating (on nitride-based)

Avinit nitride-based multicomponent multi-layer coatings were deposited in accordance with the proven process schemes.

Ti-Al 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.

Coatings (Ti-Al-N) is an example of a multicomponent coating which, by the prevalence of use, has outdone its predecessor TiN due to its higher heat resistance, hardness, and better tribotechnical characteristics.

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 % 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 [46].

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 %.

Similar are the dependencies of microhardness on the coating composition, which are given by authors of other works [46, 47], 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 [1, 48–55].

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 [56], 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.

The hardness and strength of the layer coating increases with decreasing thickness of the individual layers [56].

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

In [4–9, 22–25], numerous experimental studies of Avinit multi-component multilayer ion-plasma coatings based on the Ti-Al-N system were performed.

Table 3.1 provides the results of experiments to determine the coatings growth rates obtained on substrates performing planetary motion.

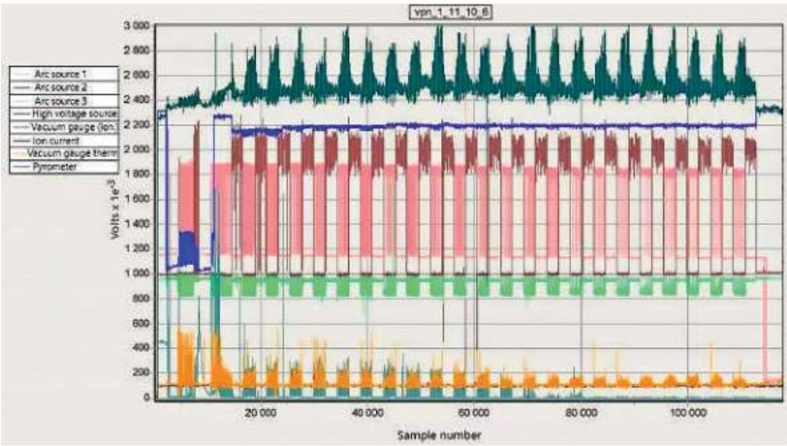
Based on the data on the coatings growth rate, data was entered into the Avinit installation control program to obtain TiN-AlN nanolayer coatings.

Table 3.1 The growth rate of different composition coatings

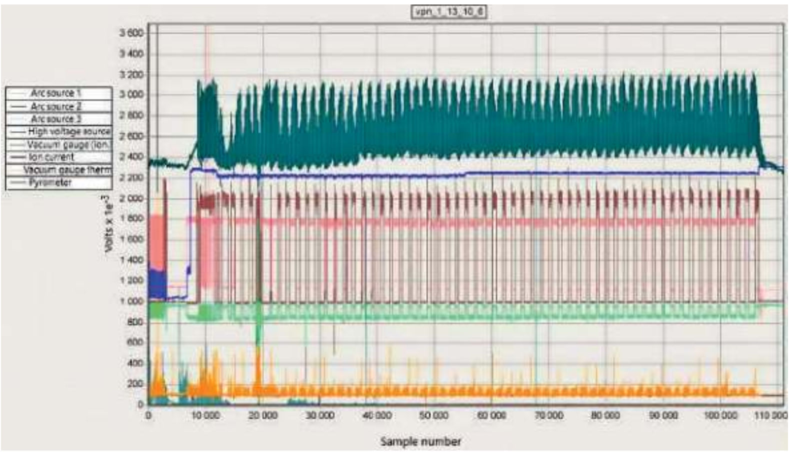
No.	Coatings	Growth rate, *V*, μm/hour	Notes
1	Avinit C300	0.7	Planetary motion
2	Avinit C310	0.25	Planetary motion
3	Avinit C320	0.25	Planetary motion
4	Avinit C350	0.30	Planetary motion

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. 3.2.



a



b

Fig. 3.2 Fragment of the automated process control system protocol of the TiN-AlN nanocoating: *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 3.2.

Table 3.2 Samples characteristics

No.	The composition	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
The coatings based on Ti-Al-N (TiN-AlN)								
1	Avinit C/P 300	59–60	Multi-layered	200	$3{\cdot}10^{-1}$	10.0	2600–3000	0.040 (11b)
2	Avinit C/P 310	59–60	Nano-layered	200	$3{\cdot}10^{-1}$	1.5	‡ 3000–3200 HV_{100} † 2500–3000 HV	0.036 (12a)
3	Avinit C/P 320	59–60	Nano-layered	200	$3{\cdot}10^{-1}$	1.5	‡ 3000–3500 HV_{100+} † 3000–3500 HV	0.036 (12a)
4	Avinit C/P 350	59–60	Nano-layered	200	$3{\cdot}10^{-1}$	1.5	2600–3500	0.036 (12a)
5	Avinit C/P 380	59–60	Nano-layered	200	$3{\cdot}10^{-1}$	3 – 5	2500–3500	0.026 (12b)
6	Avinit C/P 410	59–60	Nano-layered	200	$3{\cdot}10^{-1}$	3 – 5	1570–2550	0.025 (12b)
7	Avinit C/P 710	59–60	Nano-layered	200	$3{\cdot}10^{-1}$	3 – 5	2500–3000	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 3.2).

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 3.2).

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.

For a more detailed characterization of the structure and properties of the Avinit C310 type of nanocoatings, X-ray-based studies were performed which proved that the coatings have ~45 at. % Al in their composition.

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 (CS) 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. 3.3 shows the results of Avinit C320 coating electron-probe X-ray microanalysis (EPRMA) performed for three elements: aluminum, iron and titanium on the Avinit C320 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).

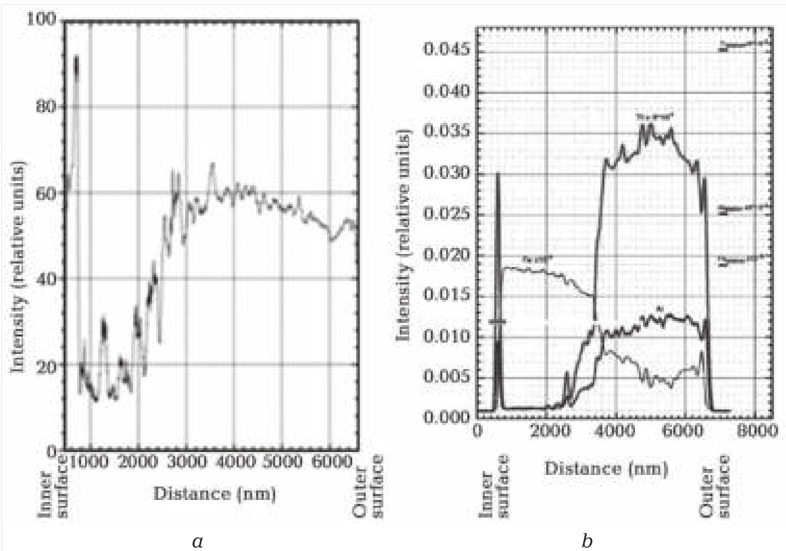


Fig. 3.3 Distribution of characteristic X-ray radiation of element atoms in Avinit C320 coating: *a* — % Ti; *b* — % Al

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.

Results of metallographic studies of the Avinit C320 coating (JSM T-300 raster electron microscope).

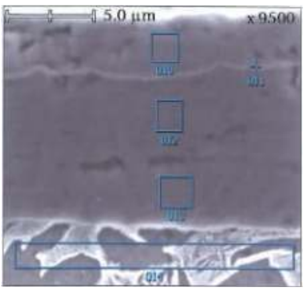
The coatings have a multilayered structure and consist of layers based on titanium, molybdenum and their compounds with nitrogen in different combinations. Microhardness $H\mu = 1.500–3.500$ MPa (depending on coating composition).

The results of metal-physical measurements of the Avinit C310 coating on a JSM T-300 scanning electron microscope are shown in Fig. 3.4–3.6.

Microhardness values of the coated specimens surfaces provided in Table 3.2, 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).

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 mN/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.



a

No.	N	Al	Ti	Mo	Total, %
010	9.10	27.93	62.96	–	100
011	6.89	16.73	76.38	–	100
012	10.7	45.87	43.44	–	100
013	10.71	42.22	42.07	–	100
014	–	3.64	88.94	7.41	100

b

Fig. 3.4 Avinit C320 coating: *a* — appearance of Avinit C320 coating (cross-section); *b* — approximate chemical composition of the analyzed areas

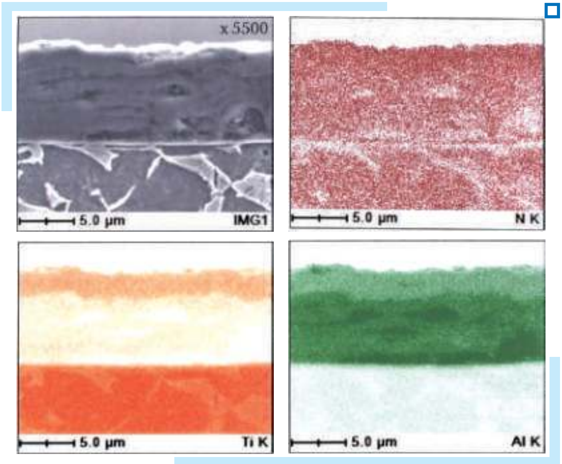


Fig. 3.5 The appearance of the Avinit C310 coating (cross-section) in the mapping mode of the coating area. The higher element content corresponds to the more intense coloring

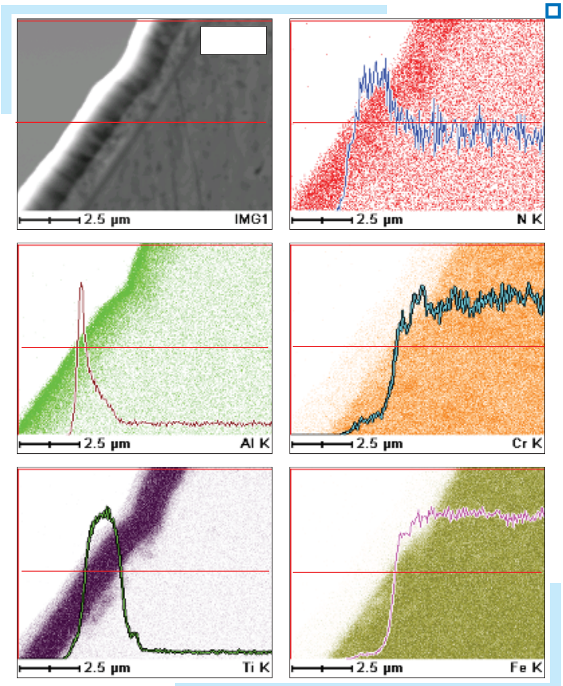


Fig. 3.6 Appearance of Avinit C310 coating in the in-line analysis mode

The measurements of microhardness and Young's modulus in Avinit C320 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. 3.7).

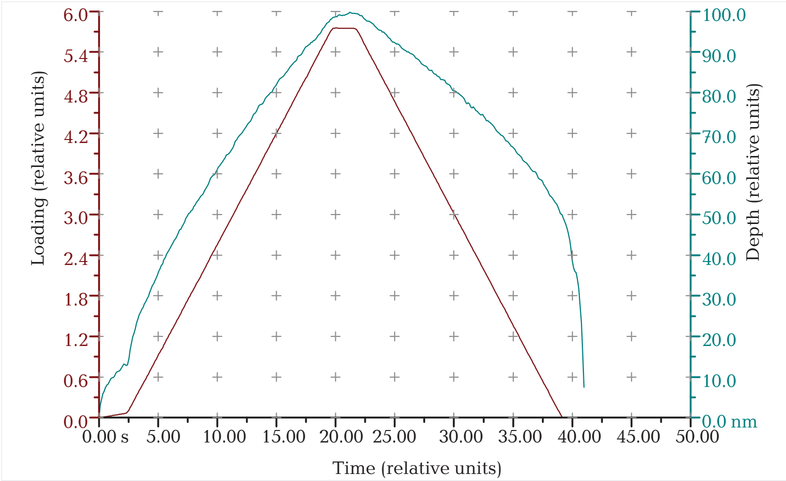


Fig. 3.7 Load diagram for Measuring Nanohardness and Young's Modulus of Avinit C320 coating

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. 3.8, *a* shows the dependences of the secondary Al^+, Ti^+ ions currents on the sputtering time for the Avinit C320 coating, and, respectively, and on the depth of the component distribution profile.

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 C310 are shown in Fig. 3.8, *b*. 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.

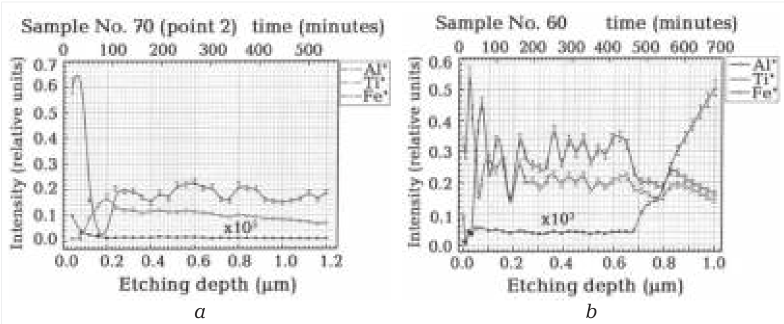


Fig. 3.8 Dependences of secondary Al^+, Ti^+ ions currents on sputtering time: *a* — Avinit C320 coating; *b* — Avinit C310 coating

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.

3.1.2 Avinit D coating (carbide-based)

When creating new designs of functional multilayer coatings with improved tribological characteristics, coatings in 'metal-carbon' systems, in particular, coatings based on Ti-C and Mo-C, with great potential for use as anti-friction wear-resistant coatings for friction couples, are of great interest.

The works [11–15] are dedicated to the development of new Avinit D multilayer coatings based on metal carbides.

We have developed new processes (PVD and hybrid PVD+CVD) of controlled formation of Avinit D multicomponent nano- and microstructural coatings in 'metal-carbon' systems using vacuum plasma (PVD) and plasma-chemical (CVD) processes of depositing multicomponent multilayer and nanolayer metal-carbon coatings (MeC, MeC:H, Me-CN, MeC-C) (Me=Ti, Mo).

Nano- and micro-layer multicomponent coatings of 'metal-carbon' systems (TiC; TiC-N, TiC-C) and (MoC, MoC-C, Mo-C-N) were selected as candidate coatings for the study.

The development of new perspective nanocoatings, it is envisages:

- creation of new anti-friction designs, wear-resistant Avinit D coatings for use in friction pair with thermally-reinforced steel and steel coated with microstructural or nanostructural coating of Avinit type;
- elaboration of antifriction wear-resistant coatings deposition on steel specimens with precision surfaces to carry out metal-physical and tribological studies;
- conducting tribological studies of 'coated steel — steel' couples;
- selection of different couples according to the results of tribological tests of optimal designs of friction couples in 'coating steel' systems and 'coating-Avinit coating type';
- selection, according to the study results, the optimal combination of the coating material for further elaboration of the selected coating on actual products.

1. Avinit D/P coating.

Elaboration of technological parameters of PVD processes for depositing multilayer and nanolayer coatings in Ti-C and Mo-C systems was performed using Avinit installation [19].

Titanium BT-1-0, molybdenum MVPCh, graphite AG-1500 were used as the cathodes materials.

To obtain the carbide-containing layers, benzene C_6H_8 was used as the reaction gas.

The composition of the residual gases and impurities in the reaction gas was monitored using a mass spectrometer MX-7304A.

With the use of upgraded PVD deposition, the processes of depositing single-layer and multilayer coatings of Avinit D/P 100 (TiC, Ti-CN, TiC-C) and Avinit D/P coatings 200 (MoC, MoC-C, Mo-CN) were elaborated.

For the deposition of multilayer coatings (TiC-Ti) and (MoC-Mo) built on the sequence of metal and carbide layers, a two-cathode scheme was used while simultaneously working one-component cathodes (Me and C) arranged towards each other in the environment of residual gases with rotation samples around their axis with continuous or pulsed (periodic) operation of sputtering sources.

Obtaining high-quality coatings using such a scheme was possible due the provision of stable controlled combustion of graphite cathode due to the modernization of cathode units and control system.

The following multilayer coatings of different composition and structure in 'metal-carbon' systems have been elaborated – monolayer TiC, monolayer TiC-N, nanolayer TiC-TiN, nanolayer TiC-C, MoC monolayer structures, monolayer Mo-C-N, nanolayer MoC-C structures to carry out studies of their properties, in particular, tribological characteristics in different friction couples (depending on the layers composition and ratio) and the possibility of their use as wear-resistant and antifriction coatings.

2. Avinit D/C coating.

Using the cross-synchronization technology to control the coating deposition process [19], we have developed new processes for the controlled formation of multicomponent nano- and microstructural coatings in 'metal-carbon' systems using vacuum plasma (PVD) and plasma chemical (CVD) PVD+CVD) processes.

Carbide-containing coatings are obtained by deposition from plasma metal fluxes (Ti, Mo, Zr, Cr, Nb) in a C_6H_8 benzene vapor environment.

For deposition of multilayer coatings of (TiC-Ti) and (MoC-Mo) types, built on the sequence of metal and carbide layers, a single-cathode scheme was used with continuous operation of the sputtering source (Ti, Mo, Zr, Cr, Nb) and pulsed (periodic) supplying the reaction gas (benzene C_6H_8 vapor).

At the same time, based on the concept of nanolayer coatings and using the capabilities of upgraded equipment, the following coatings were elaborated in the systems 'metal-carbon' – Avinit D/C 100 coatings – monolayer TiC:H, nanolayer TiC:H-TiN, nanolayer TiC-C:H, Avinit D/C 200 coatings – monolayer MoC:H, nanolayer MoC: H-TiN, nanolayer MoC:H (with different carbon content).

All studies of the deposition processes of different composition coatings were performed under conditions that did not lead to an increase in the temperature of the samples above 200 °C.

At the stage of vacuum-plasma purification, this was achieved by the use of impulse processing mode and the selection of the ratio of the operation intervals and pause of arc sources, as well as the total processing time.

When forming coatings using a titanium cathode close to the optimum, the mode of operation of the arc source was 2 seconds and 4 seconds pauses with a total processing time of 3–5 minutes with a gradual increase in the accelerating potential from 30...50 V to a maximum value of 1000...1200 V.

When working with a molybdenum cathode, the pause increased to 6 sec.

At the stage of coating deposition on the titanium basis it was possible to keep the temperature in the range of 180–200 °C in the mode of vacuum arc source continuous operation at a potential of 30–40 V.

In case of molybdenum-based carbide coatings deposition, even at 25 V, it was impossible to keep the temperature of the samples within the specified limits at constant source operation, therefore, a pulse mode of operation with a cycle of 6 seconds — work, 4 seconds — pause was used. Studies of the phase composition of coatings obtained by the deposition of titanium in benzene environment, reveal the presence of TiC phase, depending on the pressure of benzene and the coating formation conditions, among which the temperature and displacement potential have the greatest influence.

The partial pressure of benzene, at which the titanium carbide phases manifest themselves in the coatings, is at the boundary close to $1{\cdot}10^{-3}$ Pa.

In monolayer condensates deposited within the pressure range of benzene from $2{\cdot}10^{-3}$ mm Hg to $2{\cdot}10^{-2}$ mm Hg, carbon content increases from 38 % up to 52 %. This corresponds to a change in the carbon-titanium content ratio from 0.7 to 1.04.

In this case, as evidenced by the study, there is a correlation between the change in the composition of the coatings and the course of the microhardness dependence on the partial pressure of benzene, at which the coatings obtained — the microhardness changes monotonously, reaching a maximum of $H=2{\cdot}10^4$ MPa.

With further increase in the benzene pressure ($P=5{\cdot}10^{-3}$ mm·Hg) its content increases in the coating, and the microhardness increases up to $3{\cdot}10^4$ MPa.

The effect of ion energy on the coatings properties was studied on X12MF1 steel samples.

The deposition was carried out at a pressure of benzene $P=6{\cdot}10^{-3}$ mm Hg and $P=1.5{\cdot}10^{-3}$ mm·Hg and values of the accelerating potential from –50 V to –300 V.

The temperature of the samples varied from 200 °C to 600 °C.

Such changes in the deposition conditions virtually do not affect the microhardness of the coatings obtained.

At the same time, the deposition modes, in particular, the temperature and the condensation time, significantly affect the condition of the substrate material.

Virtually in all modes that provide high quality coatings, the steel DIN 1.2379 temper occurs and the more, the higher the energy of ions during condensation and deposition time in the conditions of the sample rotation around the axis in the rotary device to obtain uniform thickness of the coatings.

Studies of the coated samples roughness proved that its value is determined by both the roughness of the initial surface of the substrate and the coating deposition modes.

When treating the original surface to a purity corresponding to grade 6–7, coating deposition did not reduce the purity grade and subject to the deposition mode could even increase it by several units.

Thus, after coating deposition on the original surface, in the mode of forming a layered structure, of the steel sample treated to purity grade 7, the value of the roughness R_a had values corresponding to purity grade 10.

Reduction in the thermal load during condensation due to reducing the density of the ion current, the use of a straightline separator to reduce the droplet phase, increase the uniformity of the coating structure and ensure the preservation of the surface purity grade at the level of V13.

The study of the molybdenum-based carbide coatings deposition, as in the case of carbide titanium coatings, was carried out on quenched steel with low tempering temperature, polished to V12 purity grade.

The coatings were obtained at an ion current density of I=10 mA/cm^2, accelerating the potential of the substrate U_n=–25 V, and the temperature of the substrate T_n<200 °C.

Benzene pressure during condensation 2.5·10^{-3} mm·Hg.

When depositing carbide-molybdenum coatings without the use of separating devices, the microhardness of the coatings having thickness of 12 μm equals to 2500 kg/mm^2 and with an increase in substrate temperature, is reduced to 2.000 kg/mm^2.

The roughness of the coatings surface corresponds to the V 7–8 class when deposited on a polished to V12 purity grade surface of steel DIN 1.2379. The condensation process at temperatures not exceeding 150–200 °C does not lead to a significant decrease in the hardness of X12MF1 steel initial surface.

The regularities of changes in surface morphology (roughness) which are caused by ion etching and coating deposition 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 hardness both at the stage of ion purification and condensation.

With increasing pressure of benzene during condensation, there is virtually no morphological changes and microhardness of coatings obtained at different values of the parameters U_n (–15...–70) V and T_n (150...500 °C). Increasing the ions (U_n) energy during deposition is similar to the effect of temperature and helps to obtain structurally more homogeneous coatings.

According to the studies performed, for the formation of quality coatings based on Ti, the pressure of benzene should be maintained at 3...4·10^{-1} Pa, and for compounds based on Mo — 1...2.5·10^{-1} Pa. The obtained coatings were studied (microslice, coating hardness, determination of surface geometry after coating).

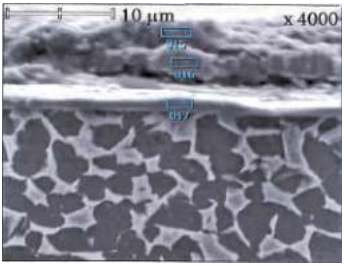
Characteristics of coatings are given in Table 3.3.

Metal-physical measurements of Avinit D/P 100 and Avinit D/P 200 coatings were performed using a JSM T-300 scanning electron microscope (Fig. 3.9–3.11).

A transverse static fracture of the coated parts was made to measure the thickness. The coating thickness is ~6–9 microns. According to the measurements, the carbon content in coating ~10–15 %, which was fairly evenly distributed in the structure of the coating in accordance with its design. Studies indicate high quality of adhesion — no detachment of the coating from the base on all investigated coatings was detected.

Table 3.3 Characteristics of coatings

No.	Samples	Microhardness, H_v, kgf/mm^2	Thickness h, µm	Roughness R_a, µm
1	Avinit D/P 100-µ1 based on Ti-C	2300	1...2	0.036
2	Avinit D/P 100 based on Ti-C	2500	12	1.45–1.15
3	Avinit D/P 100 based on Ti-C	2500	8–10	–
4	Avinit D/P 100-t10/5 based on Ti-C	2500	12	–
5	Avinit D/P 100-t10/5 based on Ti-C	2300	8...10	–
6	Avinit D/P 100-t10/5 based on Ti-C	2000	8...10	–
7	Avinit D/P 100-t10 based on Ti-C:H	2500	8...10	–
8	Avinit D/C 100-µ1 based on Ti-C:H	1700	1.5	–
9	Avinit D/C 100-µ1 based on T-C:H	2000	1	–
10	Avinit D/P 200 based on (Mo-C)	1800	1	–
11	Avinit D/P 200 based on (Mo-C)	2500	10	–
12	Avinit D/P 210 based on (Mo-C)	2200	1	–
13	Avinit D/P 200 based on (Mo-C)	2200	8–10	1.15–1.05
14	Avinit D/C 230 based on Mo-C:H	2300	1.5	–
15	Avinit D/C 240based on Mo-C:H	2500	8–10	1.15–1.05
16	Avinit C/P 100 based on Ti-N	2500	12	1.45–1.15
17	Avinit C/P 110 based on TiN	2000	10	–
18	Avinit C/P 210 based on Mo-N	2200	1.5	–
19	Avinit C/P 330 based on Ti-Al-N	3500	1.5	–
20	Avinit D/P 130 based on Ti-TiC-TiN	–	–	–
21	Avinit D/P 120-t10 based on Ti-C-C	–	–	–
22	Avinit D/C 120-n1 based on Ti-C:H	2000	1	–
23	Avinit D/P 100-t10 based on Ti-C	2500	12	1.45–1.15
24	Avinit D/P 200 based on Mo-C	2300	8–10	1.15–1.05
25	Avinit C/P 210-µ1 based on Mo-N	2300	1–2	0.44
26	Avinit C/P 330-n1 based on Ti-Al-N	–	–	–
27	Avinit C/P 110-t10 based on Ti-N	–	–	–



No.	C	Ti	Total, %
015	12.72	87.28	100
016	6.54	93.46	100
017	5.02	94.98	100

a *b*

Fig. 3.9 The appearance of the Avinit D/P 100 coating (Ti-C system-based) (cross-section) with the indicated areas of analysis — *a*; approximate chemical composition of the analyzed areas — *b*. The coating thickness is ~3.5 µm

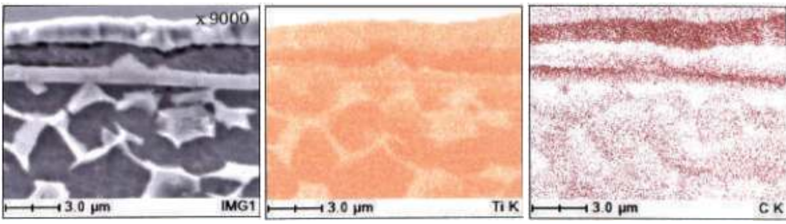


Fig. 3.10 Appearance of Avinit D/P 100 coating (Ti-C system based) (cross-section) in the mapping mode of the coating area. The higher element content corresponds to the more intense coloring. The coating thickness is ~3.5 µm

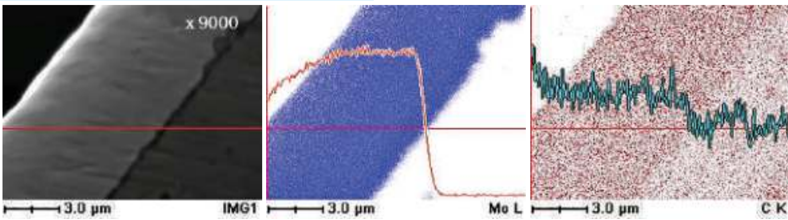


Fig. 3.11 Appearance of Avinit D/P 200 coating (Mo-C system based) in the in-line analysis mode. The coating thickness is ~6 µm

The studies performed on coatings of different composition show that the elaborated modes allow to obtain quality coatings with high adhesion, maintaining the substrate hardness — steel DIN 1.2379 within the specified limits — the hardness and microhardness of the substrate material in the selected modes of coating is virtually not reduced compared with the original state. There were no cases of peeling of the coatings when applying the scratch mesh. Metallographic studies of the coated samples confirmed the consistency and uniformity of the coating thickness over the entire surface of the samples.

The profilographic measurements confirmed that after coating deposition using separating devices on test samples with a roughness corresponding to the 12–13 purity grade, the surface roughness of the samples virtually does not change, or the surface purity grade decreases slightly (by one or two units).

Thus, using the technology of cross-synchronization control of the coating deposition process, new processes (PVD and hybrid PVD+CVD) of controlled formation of Avinit D multicomponent nano- and microstructural coatings in 'metal-carbon' systems using vacuum-plasma (PVD) and plasma-chemical (CVD) processes are developed.

Based on the concept of nanolayer coatings, the technological parameters were elaborated for the processes (PVD and hybrid PVD+CVD) of multilayer and nanolayer coating deposition (MeC, MeC:H, Me-C-N, MeC-C) (with different carbon content).

The following processes have been elaborated and coatings obtained in 'metal-carbon' systems:

a) in PVD processes:

- TiC monolayer, TiC-N monolayer, TiC-TiN nanolayer, TiC-C nanolayer;
- MoC monolayer, Mo-C-N monolayer, MoC-C nanolayer coatings;

b) in hybrid PVD+CVD processes:

- TiC:H monolayer, TiC;
- TiC:H nanolayer, TiC-C:H nanolayer;
- MoC:H monolayer, MoC:H-TiN nanolayer, MoC:H nanolayer coatings.

Optimization of depositing high-quality bonded hardening and antifriction coatings on the samples under study was carried out. Measured characteristics of coatings (microhardness, hardness, roughness).

Metallographic studies confirm the possibility of low-temperature deposition of high-quality, wear-resistant Avinit D metal-carbon-based coatings in the developed nanolayer coating deposition processes, while providing good adhesion to substrate materials (DIN 1.2379 steel) and without reducing the steel strength (<200 °C) and without degradation of the original surface purity grade.

3.1.3 Avinit V coating properties

In work [10] the process of metal coating process by gas-phase deposition using organometallic compounds (chromium, molybdenum, tungsten carbonyls) was studied, in particular, Mo coatings by thermal decomposition of molybdenum hexacarbonyl $Mo(CO)_6$ into complex profile precision surfaces.

The coatings were applied to pre-heat-treated DIN 1.2379 and DIN 1.773 steels of technical purpose with high-grade purity surface treatment (>10).

These steels are widely used in aircraft construction, and improvement of their design properties, eventually, leads to a significant increase in the service life of aircraft units.

Process conditions take into account the possibility of widespread adoption of coating deposition technology in the industry.

The development of Mo coating processes was carried out by thermal decomposition of a metal-containing compound – $Mo(CO)_6$ molybdenum hexacarbonyl on the Avinit V gas-phase unit designed for carrying out CVD processes. Mo coatings were also deposited on the internal calibrated surfaces of 12×20 tubes.

The surface purity grade is 9*a*.

The feature of the samples preparation is that their preliminary heat treatment was carried out in full accordance with the technological regulation of finished products processing accepted in production.

According to the studies conducted, this significantly affects the quality of the coating obtained.

The studies were carried out at temperatures of 350–450 °C.

This temperature interval is chosen as the optimum, since at higher temperatures there is a temper of steel, which dramatically reduces the performance of the final product and is unacceptable for industrial applications.

The technological data of the coating process on steel DIN 1.773 and DIN 1.2379 is presented in Table 3.4.

Table 3.4 Mo coating obtained when releasing carbonyl from the container

T, °C	*P*, Pa	τ, min	δ, μm	*V*, μm/min	Adhesion
350	5.20	10	8	0.80	+ + +
	10.00	10	7	0.70	+ + +
	5.30	15	17	1.13	+ + +
	11.00	15	10	0.67	+ + +
	8.80	30	25	0.83	+ + +
	11.00	30	31	1.03	+ +
400	5.60	5	3	0.60	+ +
	5.40	10	8	0.80	+
	5.50	10	6	0.60	+
	6.10	10	8	0.80	+
	5.00	15	10	0.67	+
	5.40	15	12	0.80	+
450	7.60	5	6	1.20	+ + +
	5.30	15	17	1.13	+ + +
	7.20	15	20	1.33	+ + +
	5.10	30	12	0.40	+ + +

+ + + – coating is removed by etching only; + + – minor chips; + – multiple chips

Table 3.5 presents CVD-process parameters of Mo coating deposition on X12F1 steel samples.

Table 3.5 The parameters of the CVD coating process

No.	Temperature, °C	Time, min	Microhardness H_v, kgf/mm^2	Thickness, δ, μm
23	430	10	2500	15
25	360	10	1800	10
26	290	10	2500	10

The properties of coatings differ sharply within a given temperature range.

At 350 °C and 450 °C a stable uniform coating deposition with a high value of microhardness is observed: H_v=2200 at 350 °C, H_v=1700 at 450 °C.

The thickness of the coating is linearly dependent on the dwell time.

At 450 °C, the coating has good adhesion to the substrate, at a lower temperature, poor adhesion to the original sample is observed.

Changing operating pressure within 0.01...0.1 Torr manifests at the carbon content change in the Mo coating and results in a slight decrease in adhesion, which becomes more noticeable as the coating thickness increases.

The microhardness of the obtained Mo coatings H_v=1700...2200.

This is due to the high carbon content in the coatings.

To carry out metallographic studies, molybdenum coating was performed via with gas-phase deposition on samples in cubes having dimensions ~10×10×10 mm in different conditions. After coating deposition, the surface of the samples was subjected to galvanizing.

Measurement of the coating microhardness was carried out on the hardness tester from 'Leco AMH 43', load 100 g.

The values of hardness measurements are presented in Table 3.6.

The X-ray studies reveal a significant amount of Mo_2C carbide phase which accounts for the increased microhardness of the Mo coatings.

As shown in work [57], at higher temperatures (surface temperature above 500–700 °C, P=0.1 mm·Hg), Mo coatings with substantially lower carbon content (up to 0.11 weight %) can be obtained, which causes a much lower microhardness of the obtained Mo coatings HB_{100}=400 and even less.

Table 3.6 The values of hardness measurements

No.	Microhardness min $HV_{0.1}$	Microhardness max $HV_{0.1}$	Microhardness average $HV_{0.1}$
48	1588	1945	1788
55	1122	1279	1190
62	1296	1407	1333
64	1474	1734	1660
65	1014	1242	1108

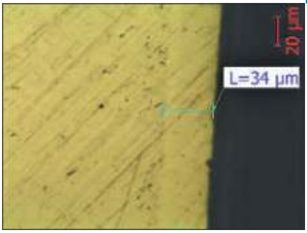


Fig. 3.12 Microstructure of Mo coatings

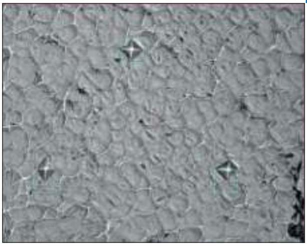


Fig. 3.13 Prints of microhardness measurements of Mo coatings

Therefore, the coatings obtained in work [10] should probably be called molybdenum-carbide coatings or, by analogy with 'hard' electrolytic chromium — 'hard' gas-phase molybdenum.

The rate of coatings deposition obtained in the atmosphere of residual vapor at a temperature of 350 °C, is uniform at sufficiently long time intervals.

The average coating deposition rate is 50...70 μm/h.

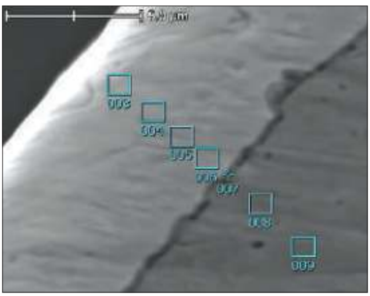
Technologies of multi-stage processes were elaborated when carrying out 3...5 technological cycles to obtain thick coatings.

The coating thickness obtained is 100 μm. The coating delamination was not observed. However, the quality of the coating deteriorates as the thickness of the coating increases, which is obviously due to the accumulation of large internal stresses in the film, the removal of which requires normalizing annealing.

Metal-physical studies of the samples obtained were performed using a JSM T-300 scanning electron microscope.

Fig. 3.14 shows the appearance of Mo coating on DIN 1.2379 steel samples (cross-section).

The appearance of Mo coating on DIN 1.2379 steel samples in the mapping mode provided in Fig. 3.15.



a

No.	Si	Cr	Fe	Ni	Mo	C
003	–	–	–	–	97.0	3,0
004	–	–	–	–	96.5	3,5
005	–	–	2.21	–	94.79	3,0
006	–	–	3.17	–	94.83	2,0
007	–	10.13	22.67	9.90	55.31	2,0
008	0.21	6.85	92.13	–	–	0,8
009	0.34	7.13	91.73	–	–	0,8

b

Fig. 3.14 The appearance of Mo coating on DIN 1.2379 steel samples with indicated analysis areas — *a* and chemical composition of analyzed areas — *b*

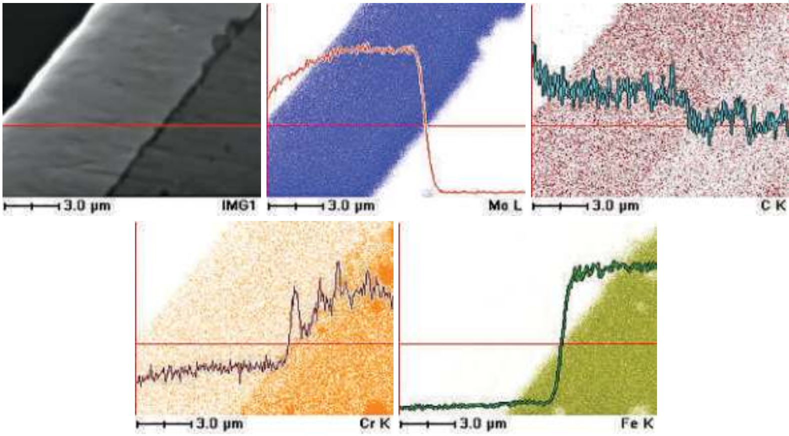


Fig. 3.15 The appearance of Mo coating on DIN 1.2379 steel samples in the mapping mode. The higher element content corresponds to the more intense coloring

Metal-physical studies proved a rather high degree of coincidence of the phase composition of the base material — DIN 1.2379 and DIN 1.773 steels (EI10) (areas 009 and 029, respectively). Fig. 3.16, 3.17 shows photos of the microrelief of the coatings surface.

Thus, a process of coating deposition 'hard' molybdenum (molybdenum carbide coatings with high carbon content) by pyrolysis of carbonyl molybdenum was developed using a gas-phase unit of the Avinit installation.

The processes of depositing high-quality bonded coatings on samples under study were optimized.

The characteristics of coatings (microhardness, phase composition, roughness, substrate hardness) were measured.

The coating deposition rate is up to 50–90 μm/h.

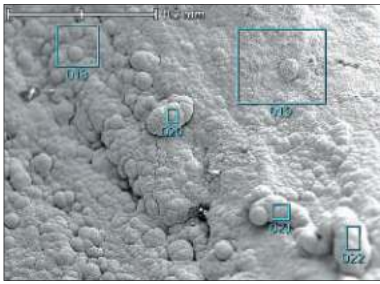


Fig. 3.16 Microrelief of the sample surface (steel DIN 1.773)

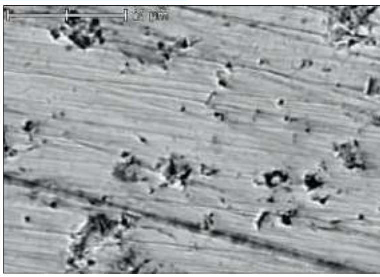


Fig. 3.17 Microrelief of the sample surface ((steel DIN 1.2379)

Metallographic studies confirm the possibility of low-temperature coating deposition of 'hard' gas-phase molybdenum in the developed CVD process, while ensuring good adhesion to the substrate materials (steel DIN 1.2379, 25X2MF) without reducing the steel strength and without deterioration of the original surface purity.

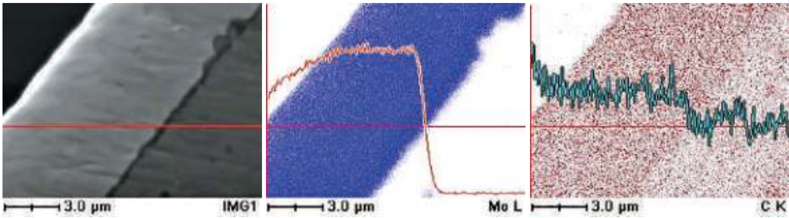


Fig. 3.18 The appearance of Mo-C coating on DIN 1.2379 steel sample in the mapping mode

The conducted research reveals high tribological characteristics of molybdenum-carbide coatings and testify to the viability of pilot-industrial technologies development for the choice of optimal coating designs for precision units of aviation units building.

The good reproducibility of the obtained coatings allows in the future to elaborate the serial production technologies.

3.1.4 Antifriction coatings based on molybdenum disulfide

When developing new design solutions, the development of advanced technologies and materials with enhanced technical characteristics are of paramount importance.

One of the important lines of work in this regard, is to improve the reliability and enhance the service life of friction units.

Due to the sharp difference in the material properties of the friction components in volume and in the thin surface layer, which determines the friction and wear parameters, it is urgent to use new technologies of depositing wear-resistant, antifriction and running-in coatings that provide extended capabilities for working layers formation [20].

MoS_2 (molybdenum disulfide) is the most common solid lubricant.

Molybdenum disulfide has an extremely low friction coefficient, lower than that of Teflon and graphite.

The achievable friction coefficient is less than 0.05, varying subject to humidity and friction conditions.

The unique properties of the molybdenum disulfide film can also account for the reduction friction coefficient with increasing speed of the relative motion in the friction pair, reducing the friction coefficient with the increase in load.

MoS_2 dry lubricants remain functional even at high temperatures. In the oxygen environment, the friction coefficient remains relatively low at temperatures up to 400 °C. In a dry oxygen-free environment, lubrication, even with oxidation products, remains stable up to 700 °C.

The work [17] presents the results of studies dedicated to elaboration of low-temperature deposition of pure molybdenum disulfide coating by magnetron sputtering.

Metallophysical studies (adhesion, hardness, microhardness, structure, composition, thickness uniformity) have been carried out.

The dependences of the growth rate of pure molybdenum disulfide and copper coatings on steel substrates were determined, depending on the argon pressure, discharge power, distance from the center of the magnetron in the radial direction and the distance to the target plane.

Under optimal targets sputtering conditions and the distance to be coated by 25–30 mm on the magnetron axis, the growth rate of the molybdenum disulfide coating is 3.2÷3.7 μm/h and the copper coating is 5.6÷6.3 μm/hour.

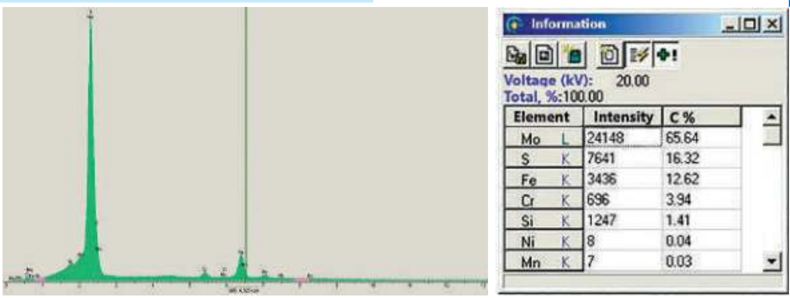


Fig. 3.19 The results of chemical composition study MoS_2 coatings

The coatings thickness can vary, ranging from 1 to 10 μm.

Coatings can be extremely thin and ultra-thin (0.1 microns or less), which is extremely important when depositing coating on precision components.

The most significant disadvantage of such coatings is their very low durability and a significant decrease in tribological characteristics in a humid environment.

To extend their scope of practical application, it is highly desirable that the coatings, while maintaining a low friction coefficient at the level of pure molybdenum disulfide, have a significantly higher wear resistance.

In works [3, 4, 6] it is shown that tribosystems based on superhard coatings (based on MoN-TiAlN have low friction coefficients less than 0.07–0.09. After testing of such friction couples for wear, no signs of high wear, bonding of the working planes of the samples with Avinit C320 and Avinit C220 coatings was detected.

Such tribosystems have already been implemented in practice [22–28].

Further improvements are associated with the creation of tribo-couples designs with even lower friction coefficients and increased durability.

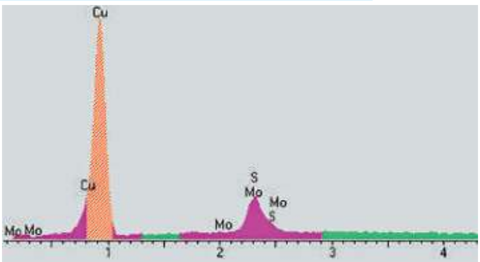
The technological capabilities of the Avinit M installation allow elaborate antifriction coatings based on the hardened molybdenum disulfide of the following type:

1. Hardened composite coatings (using combined targets).
2. Hardened nanostructural coatings (molybdenum disulfide == molybdenum nitride on low roughness surfaces followed by mechanical breaking in during operation).
3. Hardened nanolayer coatings (based on molybdenum disulfide == molybdenum, molybdenum disulfide == molybdenum nitride obtained by a combined method of vacuum-arc sputtering and magnetron sputtering).

Hardened Cu-MoS$_2$ composite coatings (using combined targets). On steel and bronze substrates of br. Su6F0,9, a batch of hardened molybdenum disulfide-coated samples was obtained by sputtering Cu+MoS$_2$ combined targets with different component ratios, at different sample temperatures and different displacement potential in argon discharge.

Fig. 3.20 presents the results of the coatings elemental composition study with coatings obtained by spraying the composite target Cu-MoS$_2$ with different ratios of components.

The studies were performed on a REM-106I scanning electron microscope with X-ray energy dispersive analysis system designed to measure the linear dimensions of topology and surface microrelief parameters of various objects in the hard phase and to measure the mass fraction of elements in the composition of objects using X-ray microanalysis.



Element		Int.	C %
S	K	2097	3.07
Fe	K	1351	4.42
Cu	K	12929	83.70
Mo	L	2180	8.81

Fig. 3.20 The results of chemical composition study coatings Cu-MoS$_2$

The coating is sufficiently homogeneous in chemical composition.

The elemental composition of samples coated with Cu+MoS$_2$ obtained by spraying the Cu-MoS$_2$ composite target onto steel substrates in different modes (coating thickness 1.8–3.0 μm) was studied by electron probe microanalysis (EPMA) (Fig. 3.21 and Table 3.7).

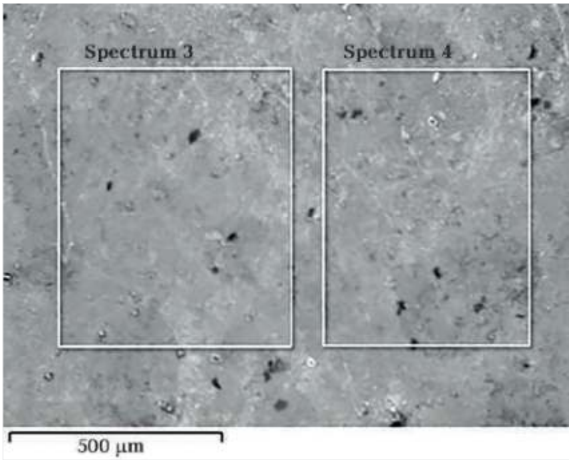


Fig. 3.21 Electron microphotography with marked X-ray spectral microanalysis areas

Table 3.7 Chemical composition

No.	Chemical composition, %			
	S	Fe	Cu	Mo
Spectrum 3	7.59	3.76	80.96	7.68
Spectrum 4	6.98	3.71	79.93	9.38

The studies' results indicated that the chosen method of sputtering of combined targets Cu + MoS_2 allows to obtain advanced hardened Avinit nanostructural antifriction coatings on the basis of molybdenum disulfide [20] with different adjustable amount of copper, molybdenum and sulfur depending on the components ratio of Cu+MoS_2 and conditions of coating sputtering.

Such coatings have a very low coefficient of friction at the level of pure molybdenum disulfide, but with significantly higher wear resistance.

3.1.5 Study of coatings friction and wear characteristics

The study of tribological characteristics of Avinit C coatings. In-depth tribological testing of improved multicomponent multilayer coatings obtained using the Avinit installation were carried out based on developed flow charts for the formation of multilayer coatings to find out the possibilities of their use as wear-resistant and antifriction coatings for further wear and units endurance testing and elaboration of coating deposition on specific parts in the field units of friction of aviation units.

The parameters of multilayer and nanolayer coatings of the Avinit type during tribological tests for scoring resistance and wear are given in Table 3.8.

Table 3.8 The parameters of coatings Avinit

No.		
1	Microlayer coatings Avinit C320	H_v=3500 kgf/mm^2, h=1–2 µm
2	Multilayer coatings Avinit C210	H_v=2300 kgf/mm^2, h=1–2 µm
3	Multilayer coatings Avinit C220	H_v=2300 kgf/mm^2, h=20 µm
4	Multilayer coatings Avinit C220	H_v=2300 kgf/mm^2, h=16 µm
5	Nanolayer coatings Avinit C320	H_v=3500 kgf/mm^2, h=1–2 µm
6	Multilayer coatings Avinit C350	H_v=3500 kgf/mm^2, h=20 µm

The values of the friction coefficient in a friction pair (Avinit C210/ Avinit C320), recorded in the processes of tests for scoring resistance and wear (in tests for wear — average), are provided in Fig. 3.22.

During the wear tests, the parameters recorded were at the same level during the whole test period, and the wear rate was characterized by 20...40 acoustic emission information units.

Nonfailure operating time of Avinit C350 ▼ 10 and Avinit C220 ▼ 10 coating under conditions: 1600N load, 500 rpm, lubrication — TC-1 fuel, according to this section amounted to 40 hours for each sample.

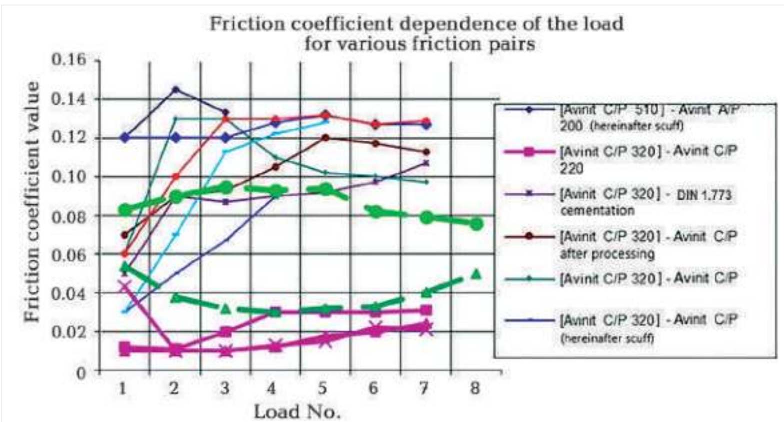


Fig. 3.22 Friction factors

Nonfailure operating time of Avinit C320 coating was 24 hours for each sample. After testing all the friction couples no signs of increased wear, bonding of the samples working surfaces with the coatings Avinit C320, Avinit C350 and Avinit C220 were observed. The maximum wear extent is in the breaking in trace area and has a value of ≈ 0.8 μm.

The study of coatings Avinit D [14, 20]. The studies were carried out for Avinit D/P 100 coatings (Ti-C; Ti-C: H, Ti-C-N, Ti-C-C) and for coatings Avinit D/P 200 (MoC, MoC-C, Mo-C-N, Mo-C: H).

Summarized dependences of the friction coefficient on the load for friction couples that worked without wear and scoring, are shown in Fig. 3.23.

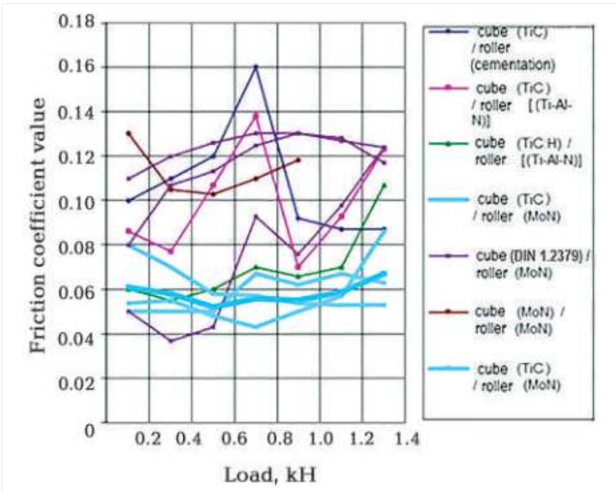


Fig. 3.23 Dependence of the friction coefficient on the load for friction couples, worked without wear and scoring

The study of Avinit M coatings. In work [20], the results of studies of hardened molybdenum-based Avinit composite antifriction coatings and the study of their tribological characteristics are presented.

Avinit M and Avinit E units were used for the coating deposition of the Avinit installation for the vacuum-arc sputtering with a large-volume vacuum chamber and an automated system for controlling the operation of vacuum-arc evaporators and the system for supplying reaction gases to the chamber working volume.

Coatings were deposited according to a preset program, which set the time, sequence and the evaporators operation mode, the reaction gas supply system, which allowed to obtain multilayer and multicomponent coatings with variable thickness composition.

When elaborating the coating processes, the main task was to select the parameters that provide a tightly bonded layers of the selected compositions without loss of strength of the base material.

Tests were performed to determine the friction coefficient and wear resistance of the Avinit C310 (TiAlN) tribosystem — MoN=10 μm+MoS_2= =5...10 μm (nonfailure operating time 16...24 h).

The friction coefficient and temperature in the contact area over time are presented in Fig. 3.24.

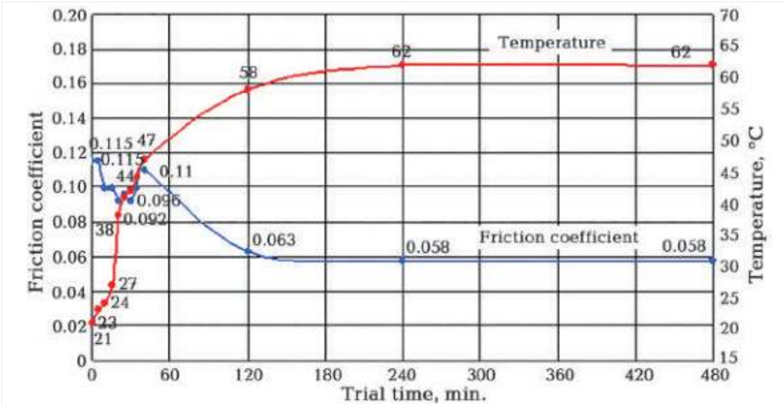


Fig. 3.24 The friction coefficient and temperature in the contact area over time

Weight wear of sample *A* — 0.0001 g, sample *B* — 0.0001 g.

The tribosystem has an abnormally low wear rate. In terms of tribotechnical characteristics and acoustic emission the tribo-pair has high performance.

The best level of antifrictionality, the tribosystem demonstrates in the range of loads from 1.000 to 1.200 N, which is probably explained by the operation of MoS_2 coating, which fills the submicroscopic irregularities of the microrelief, providing high rates of antifriction in the operational load range.

The tribosystem has a high resistance level (more than 2.000 N).

Changing the generalized acoustic emission level testifies to virtually complete tribosystem breaking in within the load range of 1.200...1.600 N.

3.1.6 Features of Avinit coatings

Summarizing the results of in-depth metalphysical and tribological studies, it is possible to formulate the features of the Avinit coatings.

The significant increase in the range of sources, which is ensured by the comprehensiveness of the methods used, allows to obtain coatings from virtually any element and alloy, refractory oxides, carbides, nitrides, metal-ceramic compositions based on refractory metals and oxides, which significantly increases the possibility of creating fundamentally new materials and coatings and parts for various purposes, operating in extreme conditions of temperature, exposure to corrosive environments, and mechanical stress.

The registration and management of the main technological parameters of the coating processes is carried out based on a special automated system.

This allows to choose the most optimal techniques and methods of surface treatment and coating or a combination of them to achieve the maximum technical and economic effect when solving specific problems.

When obtaining the Avinit coatings, it is possible to perform transition to the nanoscale for the implementation of controlled formation of multicomponent nano- and microstructural coatings with specified characteristics containing a large number of layers of different chemical composition (metal, nitride, carbide, oxide, etc.) having thickness from several units to hundreds of nanometers.

The correct choice of individual layer materials, deposition methods and optimization of technological parameters create the preconditions for the synthesis of materials with a set of unique properties, including exceptionally high hardness, strength, chemical stability, low coefficient of friction and high wear resistance.

The developed software products allow to proceed to the micro-design of functional coatings and to provide obtaining the specified nanolayer and microlayer multicomponent coatings and to reach a new level with regard to further modification and improvement of Avinit coatings designs, stability of technologies and improvement of quality control when depositing such coatings.

1. For depositing multilayer composite coatings, experimental and technological equipment has been developed and created — automated Avinit system which allows to implement comprehensive coating methods (plasma-chemical CVD, vacuum-plasma PVD (vacuum-arc, magnetron), processes of ionic saturation and ionic surface treatment) combined in one technological cycle.

To ensure implementation of the processes of multicomponent nanostructures and microstructural coatings controlled formation with controlled composition and set characteristics, a radical restructuring of the operation control of all technological equipment systems was performed on the basis of cross-synchronization technology of the systems of ion-stimulated deposition and equipment for nanoscale diagnostics of equipment due to introduction of new microprocessor power systems, synchronization and control of synthesis and diagnostics processes and development of a set of methods for technological parameters controlling during the coating deposition process to ensure purposeful process control.

The technological equipment was reconstructed via computerization of the process control, in particular, sputtering sources, control system and parameters control of the installation operation, the developed software products allowed to transfer to microconstruction of functional coatings and to provide nano- and microlayer multicomponent coatings.

These improvements made it possible to reach a new qualitative level by further modification and improvement of the Avinit-type coatings designs, technology stability and quality control when depositing such coatings for precision friction couples in machine building, unit building, and power engineering.

2. For the formation of nanostructural coatings, flow charts and their variations were elaborated depending on the type of substrate (movable or stationary), composition of nanostructural coatings and features of application with view to the requirements for providing the needed properties of coatings depending on their functional purpose.

The technological means of obtaining multicomponent metallic and non-metallic coatings by comprehensive methods of vacuum-plasma and plasma-chemical deposition from gas and vapor phase using non-equilibrium low-temperature plasma have been elaborated. The main efforts are focused on the formation of nano- and microlayer multicomponent hardening coatings as the most promising to achieve the required tribological characteristics.

3. Avinit-type coatings are deposited on high-purity grade precision surfaces up to grade 12–13 without reducing the surface purity grade.

This is achieved through the use of effective surface cleaning technologies being developed — cleaning in Ar glow discharge, cleaning in two-stage vacuum-arc discharge (TSVAD) and cleaning with metal ions at a voltage above zero growth point, as well as preventing surface damage by microarcs, for which a three-level (mechanical, electrical and electronic) arc-quenching system is provided for in the Avinit installation, that provides high quality surface cleaning for oxides and other contaminants without causing electrical breakdowns.

Deposition at low temperatures that do not exceed the temper temperature of the base material, which ensures the preservation of the mechanical characteristics of the products to be coated.

4. The Avinit coatings have a nanolayer and multilayer structure and contain a large number of layers of different chemical composition (metal, nitride, carbide, oxide, etc.) with thicknesses ranging from one to hundreds of nanometers.

Layers of different chemical composition are applied using combined methods — PVD (vacuum arc and magnetron sputtering) and CVD (gas-phase and plasma-chemical deposition).

The structure of the layers is provided by programmable harmonized modes of plasma sources operation (both PVD and CVD), working gases (argon, nitrogen, carbon and oxygen-containing gases) and high potential applied to the substrate.

At programmed changes in the amplitude and voltage pulse relative duration applied to the substrate, either above or below zero growth point (in sputter and deposition modes), large-grain columnar structures do not develop during the interrupted growth, and new centers of crystallization are generated, in which case, very fine-grained structures are implemented at deposition of even one-component metallic surfaces which ensures improvement of their mechanical and other characteristics.

5. Fundamentally new processes (PVD and hybrid PVD+CVD) of multicomponent nano- and micro-layer coatings controlled formation in metal-nitrogen and metal-carbon systems using vacuum-plasma (PVD) and plasma-chemical (CVD) processes have been developed.

The following processes have been elaborated and the coatings obtained:

a) 'hard and super hard coatings':

- on a nitride basis in 'metal-nitrogen' systems — monolayer and multilayer (Ti, Mo, Zr, Cr) N, Ti-Al-N, Ti-Mo-N, Zr-Ti-N, etc.;
- on a carbide basis in 'metal-carbon' systems — monolayer and multilayer TiC, monolayer Ti-CN, nanolayer TiC-TiN, nanolayer TiC-C, MoC monolayer structures, monolayer Mo-CN, nanolayer MoC-C, monolayer TiC:H, nanolayer TiC: H-TiN, nanolayer and multilayer TiC-C:H, monolayer MoC:H, nanolayer MoC:H-TiN, nanolayer MoC:H structures;

b) 'metal-to-metal coatings' — metal multilayer PVD coatings Mo, Ti, Zr, Nb, Cr, Ni; multilayer PVD coatings based on Cu-Mo-N; multilayer PVD coatings based on (Cu-C) (with different carbon content);

c) antifriction coatings based on molybdenum disulfide with impurities of copper, titanium nitride, etc., obtained by magnetron and CVD methods.

Characteristics of coatings Avinit CVD. Avinit A and Avinit D — specially designed series of coatings for the aerospace and aerospace industries. They represent a family of low temperature CVD (gas phase deposition), metal (Me-Cr, Mo, W) and metal carbide (Me-C) coatings that extend the life of critical metal components that work in abrasive, erosive and chemically aggressive environments. The coatings have a complex of tribological and erosion properties and chemical resistance, high hardness and workability.

Avinit A and Avinit D are a direct replacement for solid electroplating. Coatings outperform solid chromium plating in corrosion resistance, wear resistance and durability in harsh environments.

The developed Avinit A and Avinit D coatings can be deposited on elements of complex configuration and significant length, as well as on the internal surfaces of the parts.

Avinit A and Avinit D coatings are characterized by the following parameters:

- the coatings have exceptional strength, elasticity and impact resistance. They withstand deformation and shock loads well enough;

- the microhardness of metal coatings at the level of 400–800 kg/mm^2, the microhardness of multilayer metal-carbide coatings is substantially higher — $H_v \geq 1.200–1.800$ kg/mm^2);
- the coatings have excellent adhesion to the substrate, high adhesion to a wide range of materials, low friction coefficients and are resist friction scoring well;
- the porosity of the coatings is very low ~0.04 %. It is possible to obtain metal coatings of almost theoretical density;
- the erosion resistance of the coatings is 30 % higher than that of widely used stelite, and unlike stelite, it does not require finishing;
- the coatings have very high chemical resistance. The products coated resistance to resistance 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 operational level in the conditions of corrosive environment;
- the coating formation process does not adversely affect the mechanical properties and structure of structural materials;
- the endurance limit of coated steel increases by 15–25 %.

The coatings are well processed. Finishing operations are permissible — polishing, grinding, honing.

The entire array of tribological data for the coatings under study in the averaged form is presented in Fig. 3.25.

To perform the analysis of the tribological studies results, the results of 'basic' tests are provided for wear and scoring resistance of traditional friction pair [bronze br. Su3H3C3S20F0.2/nitriding steel DIN 1.7361], which are made in identical conditions. Comparison with the test results of 'coated steel — bronze br. 010S2H3, treated under IT 25.60-2003', as well as bronzes br. 010S2H3, br. Su3H3C3S20F0.2, br. Su6F0.9 with uncoated steel, indicates that the presence of developed coatings significantly increases the durability of tribo-couples to scoring, while increasing the scoring development R_{cr} value and virtually preventing scoring development.

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 C110 of TiN-Ti type) leads to an increase in P_{cr} compared to monolayer coatings (e. g., Avinit C100 of TiN-Ti type).

Particularly, effective coatings are those based on Avinit C220 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.

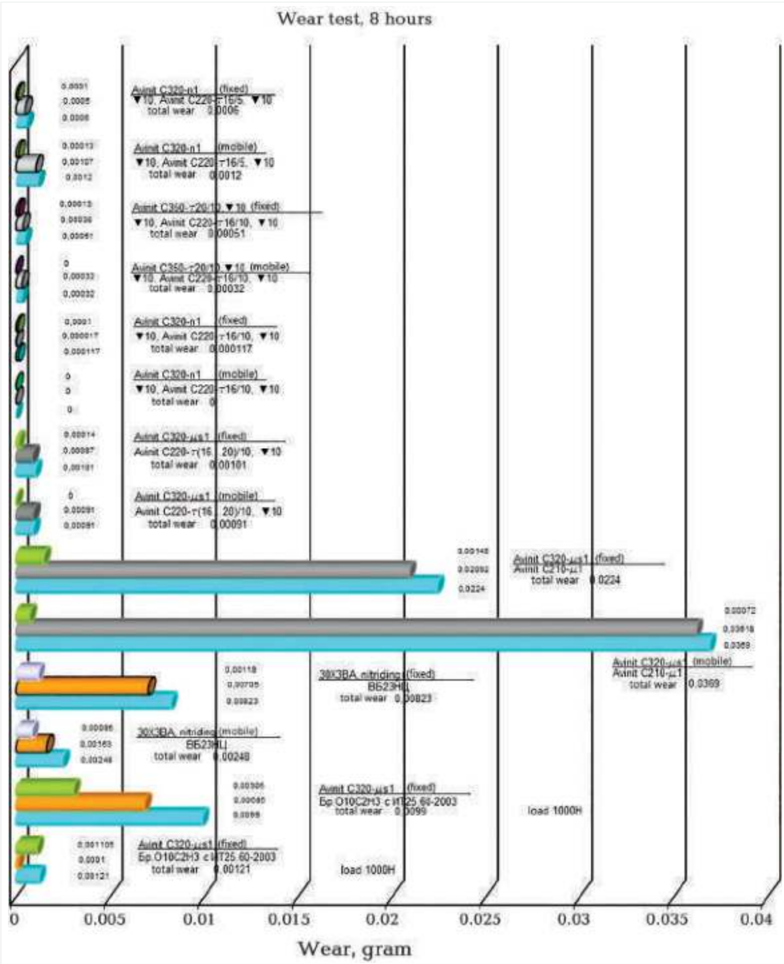


Fig. 3.25 Generalized tribological test results

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 C320 coating — Avinit C220 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 tribo-couples with the coatings under study.

All coatings in the tests showed high resistance to wear, the value of which did not exceed 0.8 microns.

Friction tribo-couples, the working surfaces of which have micro- and nanolayer coatings of Avinit C310, Avinit C320, Avinit C350, Avinit C220, Avinit C220, Avinit C220, tested under limit lubrication conditions (working fluid — aviation fuel TS-1), are characterized by:

- high resistance to scoring development;
- lack of secondary breaking in;
- rather high stability over time of the friction coefficient at operation under constant load;
- a significantly smaller difference between the 'straight' and 'reverse' couples, compared with the 'base' pair of bronze br. Su3H3C3S20F0.2/ nitrided steel DIN 1.7361.

All tested friction pairs with nanocoating have a clearly expressed breaking in working period ≈60 min, after which the values of the friction coefficients stabilize and, at constant load of 1.600 N, are within the range of 0.09...0.132.

Comparison of the bronze surface after testing with different types of coatings indicated that Avinit C320 coatings provide the best breaking in of the roller among the tested friction tribo-couples.

On the other hand, the extent of this coating type wear has minimal values, which, in general, allows to consider this couple as the best according to the results of the tests.

The best combination of durability and tribological properties was demonstrated by the friction pair composed of Avinit C220 coatings (16...20 μm thick), followed by grinding to 10...15 μm thick, and Avinit C320 (1...2 μm thick), without further machining.

This pair had the lowest friction coefficient (K_f=0.095 within the entire load range, and at maximum load of K_f=0.075) and virtually zero wear for 8 test hours.

Wear resistance significantly exceeds VB23NTs/30X3VA nitrided steel (chosen as one of the best options currently available for friction tribo-couples in aviation fuel):

a) 12 times minimum for the couple as a whole;

b) 2.5 times the minimum for a harder sample of the couple;

c) 44 times the minimum for a softer sample of the couple;

d) weight wear was not detected the applied control methods on the 'straight' couples after the tests for 8 hours.

Avinit C220, ▼ 10/Avinit C350, ▼ 10 friction couple use is expedient, at high tribological characteristics, when machining of the both parts comprising the couple can be performed after coating deposition.

The Avinit C220, ▼ 10/Avinit C320 friction couple revealed in this work the longest period of breaking in to stabilize the friction coefficients at constant load.

Weight wear detected after 8 hours of wear testing is less than that of the 'base' couple:

- by 2.7 times for 'straight' couple;
- by 8.1 times for 'reverse' couple.

Avinit C220, ▼ 10/Avinit C320 friction couple revealed in this work a complete coincidence of the friction coefficients of the 'direct' and 'reverse' couples during continuous operation under constant load.

Due to the thickness reduction of the Avinit C220 coating by machining to 5 microns, the couple wear resistance was reduced, namely: the weight wear detected after 8 hours of wear testing is less than that of the 'base' couple:

- for 'reverse' couple — by at least 1.7 times;
- for 'straight' couple — by at least 1.3 times.

The results of tribological tests indicate that carbide-based coatings of the Avinit D/P type are promising for improving the wear resistance and reducing the friction coefficient and may be recommended for further wear and units testing and for further elaboration of the coating deposition on specific field friction units.

On the basis of the performed tribological tests, a selection of advanced coatings based on the Ti-Al-N system was made to increase the wear resistance and reduce the friction coefficient of the couples:

- Avinit C/P 320-n1 coating, with a hardness of 3500 HV and a thickness of 0.001...0.002 mm, deposited on a 30X3VA nitrided steel surface and Avinit C/P 220-t20/10, ▼ 10 coating of 10...15 microns thick, applied to polished surface with a roughness of R_a 0.63 μm (▼ 8 grade);
- Avinit C/P 320-n1 coating, with a hardness of 3500 HV and a thickness of 0.001...0.002 mm, deposited on the work surface of nitrided steel 30X3VA and bronze treated as per IT.
- Avinit C/P 300 coating, with a hardness of 3500 HV, deposited on a 30X3VA nitrided steel surface and Avinit C/P 200 coating — Avinit C/P 320-ms1 coating, with a hardness of 3500 HV and a thickness of 0.001...0.002 μm.

The best combination of durability and tribological properties was demonstrated by the friction couple composed of Avinit C/P 220-t16/10 coatings (16...20 μm thick), followed by grinding to thickness 10...15 μm, and Avinit C/P 320-n1 (thickness — 1...2 μm), without any further machining.

It had the lowest friction coefficient and virtually zero wear for testing 8 hours.

On the basis of the tribological tests performed, a conclusion was made on promising nature of the following developed Avinit multi-component multilayer coating materials based on carbide systems: Avinit D/P 100 (Ti-C; Ti-C: H, Ti-CN, Ti-CC) and Avinit D/P 200 (Mo-C, MoC-C, Mo-CN, Mo-C: H) — coatings to increase durability and reduce friction coefficient.

These coatings are recommended for further wear and units testing and for the subsequent possible elaboration of the coating processes for specific details in the field friction units to carry out endurance and units tests.

Tribological tests using the method of acoustic emission and profilographic measurements proved that the improved Avinit antifriction hardened composite coatings $MoN\text{-}MoS_2$ have high tribological characteristics when working in couples with superhard coatings based on Ti-Al-N.

In the process of wearing away during operation, keeping low values of friction coefficients close to MoS_2, they have much higher wear resistance, which is many times higher than MoS_2 resistance, and significantly more stable friction coefficients, which is explained by the work of the MoS_2 coating, which fills the submicroscopic irregularities of the microrelief providing high rates of antifriction in the operational loads range.

Fig. 3.26 Development of new solutions with colleagues

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.

3.2 Application of Avinit nanocoatings to enhance performance characteristics of parts produced by unit- and engine-engineering industries

Avinit nanocoatings are very effective when used to improve the performance of various parts of the unit and engine engineering which work to wear and fatigue, in corrosive environments, etc.

The strategic line of scientific and technological development is the creation of new materials and industrial technologies via combined methods of ion-stimulating deposition and radiation modification, arc and plasma-chemical methods for the deposition of multilayer and nanolayer ion-plasma and plasma-chemical coatings and their introduction into serial production of new aviation products for gradual increase of service life and reliability of these products through the use of developed nanotechnologies.

Performed equipment improvements made it possible to reach a new qualitative level in further modification of Avinit type coatings designs, stability of technologies and increasing their quality control during the deposition of such coatings to be used in friction couples parts.

Experimental and technological developments which were performed at the new experimental and technological base, have led to the creation of a number of advanced designs of Avinit multilayer and nanolayer coatings and technologies for their production.

Research and industrial technologies for depositing such advanced coatings on specific parts of the serial units have been elaborated.

When elaborating the processes of hardening and antifriction coatings deposition to improve the wear resistance of the working surfaces of the friction-sliding couples we proceeded from the following basic requirements for the coating deposition technology:

- deposition processes must ensure reliable adhesion of the coating to the base material;
- deposition processes should not reduce the hardness of the substrate below the level indicated in the drawings on the part to be covered, increase in the surface roughness by more than 0.08 microns and curvature of the original surface by more than 0.002 mm.

Since the tempering temperature of the steels used is usually about 200 °C, the temperature of the coating deposition process should not exceed 200 °C; processes elaborated must ensure the stability of the coating deposition technology and the effective quality control; with further modification, the developed processes and technologies should become the basis of industrial technologies for the deposition of selected coatings on serial products.

After the selection of the best coating materials for friction couples, in accordance with tribological tests, coating deposition technologies were elaborated for machines specific units and serial parts of units were obtained for field resource tests.

Positive test results of friction couples were obtained, the parts of which were deposited with Avinit type coatings using nanotechnologies. In comparison with traditional friction couples, the wear resistance increases by 8 times or more, while the friction coefficients do not alter.

This makes it possible to significantly increase the durability and reliability of the fuel and hydraulic systems units of aviation engines and aircrafts.

The development and implementation of Avinit-coated 'steel-to-steel' friction couples in the design of modified NR-3VN units makes it possible to increase their service lives by 2–3 times.

According to the elaborated experimental and industrial technologies, advanced coatings of the Avinit type were deposited on specific friction couples parts of pumps and regulators of fueling units and of aircraft engines controls for carrying out units tests and final selection of coating materials for serial deposition on friction couples parts.

Friction couples, the working surfaces of which have Avinit C310 nanolayers, were tested in conditions boundary lubrication (working fluid — aviation fuel TS-1), are characterized by:

1) high resistance to scoring development; the presence of Avinit C310 coatings in tribocontact significantly increases the resistance of tribo-couples to scoring, increasing the scoring development value of RKR and virtually preventing scoring development;

2) the best breaking in of work surfaces — 'Avinit C310-bronze br. 010S2H3 coating, processed using factory technology' and lack of secondary breaking in.

The duration of the wearing away period ≈60 minutes, after which the values of the friction coefficients stabilize and, with a constant load of 1.600 N, are within the range of 0.09...0.1;

3) the best wear resistance of the both working surfaces in the absence of adhesion — 'Avinit C310-bronze coating br. Su6F0.9', processed using factory technology.

The wear resistance of Avinit C310 friction couples — bronze significantly exceeds the 'base' couple 'DIN 1.2379 — bronze br. Su3H3C3S20F0.2', currently better when working in aviation fuel.

Weight wear detected after 8 hours of wear testing:

- at least by 12 times less than the 'base' couple as a whole;
- at least by 2.5 times more for a harder sample of the couple;
- at least by 44 times for a softer sample of the couple;
- on 'direct' couples after 8 hours of weight wear tests of the coating indicated very high resistance to wear, displaying almost zero wear, which is not detected by the control methods used;
- friction coefficients lower than 'base' couple DIN 1.2379 — bronze br. Su3H3C3S20F0.2' — Avinit C310 — bronze br. Su6F0.9, processed using the factory technology.

The lowest friction coefficient small couple — Avinit C320, coating with a hardness of 3.500 HV and a thickness of 0.001...0.002 mm, deposited on the ground surface of nitrided steel DIN 1.7361 with a roughness R_a ▼ 10 without any further machining.

Avinit C310 surface preparation for coating, namely:

- grinding, with the formation of a purity grade ▼ 8;
- lapping, with the formation of a purity grade ▼ 10,

no significant differences for the test results of friction couples that include Avinit C320) were observed.

The value of the friction coefficient of the couple did not exceed 0.095 within the entire load range, and at the maximum load it equaled 0.065, which corresponds to the minimum value obtained for the friction couples with the coatings under study:

- rather high stability of the friction coefficient over time when working at constant load;
- significantly less difference between the 'direct' and 'reverse' couples, compared to the 'base' couple of bronze br. Su3H3C3S20F0.2/nitrided steel DIN 1.7361.

The above data, as well as the fact that bronze br. Su6F0.9 is extremely non-technological in diffusion welding, which is used in serial processes of friction couples manufacturing, allows to choose a friction couple 'Avinit C310 coating-bronze br. 010S2H3', processed using factory technology as the most promising for the use in the considered distributor slide valves.

According to the results of units endurance tests (Table 3.9), deposition of Avinit C310 coating on the working surface of the distributor slide valve provides substantially higher pump performance.

Table 3.9 Results of units endurance tests

	Resource of work, h
Units with distributor spool without coatings	400
Avinit C310 distributor spool unit	1000
Avinit C310 distributor spool unit	4000

During the tests, another significant positive point of depositing the Avinit C nanolayer coatings was revealed.

Usually, when operating pumps using aviation fuels, products of coking of the working environment are formed in the friction zone due to high temperature, which further aggravates the friction conditions and significantly limits the friction couple durability.

According to the tests results, the use of the Avinit C320 and Avinit C310 nanolayer coatings almost completely prevents kerosene coking in unit operating modes, which improves tribo-couple performance.

Thus, the application of Avinit C nanolayer coatings at friction units with distributor slide valve provides high reliability of the work of serial and new designs of units and increases their service life by 5–20 times.

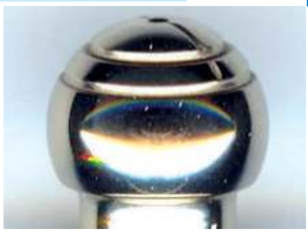


Fig. 3.27 Spherical surface of the plunger with a nanolayer Ti-based coating

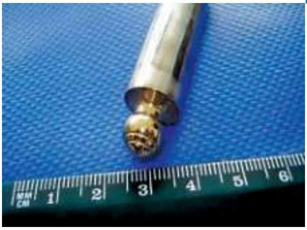


Fig. 3.28 Plunger with microlayer Mo-based coating

Increasing the service life is ensured by:

- tribological compatibility of the coating material with the material of the pumping unit at all operational modes of the pump, with some reduction in the friction coefficient;
- increasing the hardness (strength) of the working surface of the distributor slide valve up to ≥3.200 HV, the coating does not change the original geometrical parameters of the above working surface;
- eliminating the negative impact of kerosene coking products on the performance of the friction couple.

Strengthening of gas turbine engines turbocompressors blades with wear-resistant Avinit coating [29–34].

Fig. 3.29 Avinit C350 coating based on (Ti-Al-N)

Fig. 3.30 Avinit D100 coating based on Ti-C

3.3 Deposition of metal and metal-carbide coatings via CVD method on aviation units parts

The technological process of depositing composite molybdenum-carbide coatings Mo-C by the gas-phase deposition method was developed.

The processes of depositing high-quality bonded coatings for precision units of aviation units were optimized.

High performance was obtained (growth rate, quality of coatings – microhardness, phase composition, roughness, the base hardness).

Functional metal and metal-carbide coatings were developed using CVD-method for deposition on parts of aviation units with high wear resistance and tribological characteristics.

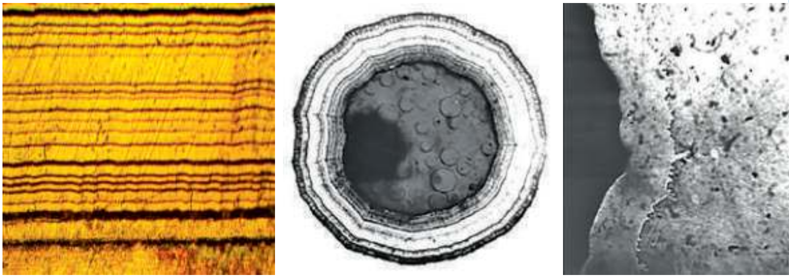


Fig. 3.31 Microstructure of molybdenum-carbide Mo-C coatings

Fig. 3.32 Aviation units stems with molybdenum-carbide Mo-C coating

The Mo-C molybdenum-carbide coatings obtained by the CVD method have a high homogeneity both in thickness and in the intersection of the stem cylindrical sections and in their length.

The coatings have sufficient adhesion, withstand the processing by polishing and grinding without compromising the integrity or delamination to ensure the required treatment purity of the coated surfaces.

The latest developed technologies provide multilayer coatings based on refractory materials with a particularly ultrafine (up to amorphous state) and ultra-multilayer coating structure with unique physical, chemical, mechanical and corrosion-proof properties.

A test was performed to determine the friction coefficients of gas-phase coatings made of hard molybdenum with nitrided steel made using the JSC FED technology 20X3MVF (disk) in the medium of T1 aviation fuel.

The tests indicated that all coatings obtained are capable of working without scoring development at loads typical for the operation of aircraft units.

When working in friction couple with the bronze SB 24 in the aviation fuel T1 environment Mo CVD-coatings have lower friction coefficients than the electrolytic coating of hard chromium, and in microhardness and wear resistance exceed it by several times.

To determine the wear resistance of Mo coatings obtained in different modes of deposition from the gas phase, tests were performed according to the Brinell (II) scheme with the flow of abrasive in the contact area at a load of 0.05 kN on a friction path of 60 m at a sliding speed of 0.78 m/s.

A 'disk' of polytetrafluoroethylene (fluoroplast-4) with a diameter of 50 mm served as a counterbody.

Quartz sand of 0.25–0.4 mm fraction was used as the abrasive.

The tribological tests carried out indicated the extent of wear, which, for comparison, was 3 to 10 times less than the wear of the electrolytic coating of hard chromium obtained by the technology used at JSC FED and tested according to the same scheme as the Mo coating.

The results obtained in the development of Mo-C coatings deposition process, are basic for the development of research-industrial technological processes of depositing such coatings on specific products of aviation and technical purpose.

3.4 Plasma precision nitriding of Avinit N steels and alloys

We have developed a method of plasma precision diffusion saturation of metals and alloys in high-density plasma [35–41].

Experiments results. The experiments were carried out on samples of widely used steels 34NiCrMoV14-5, DIN 1.773, 24CrMoV5-5.

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. 3.33, 3.34).

1. 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 [19], 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.

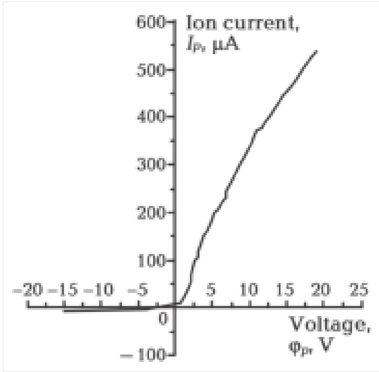


Fig. 3.33 Typical volt-ampere characteristic (VAC) of a probe at a pressure of $1.2{\cdot}10^{-2}$ Torr, using which the plasma parameters (ion current, plasma density, electron temperature, degree of flow ionization) were calculated

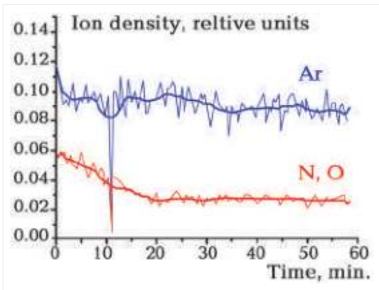


Fig. 3.34 Plasma spectral parameters

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 % N_2, 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. 3.35 and Fig. 3.36.

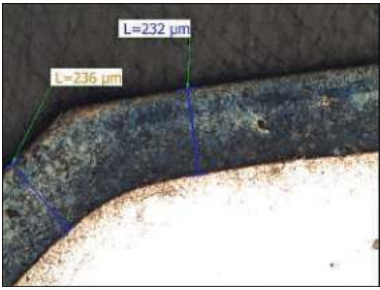


Fig. 3.35 Depth of nitrided layer on 34NiCrMoV14-5 steel (×500)

Fig. 3.36 The microstructure of the nitrided layer on 34NiCrMoV14-5 steel (×500)

The hardness value of the base metal after nitriding does not change *H*µ=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. 3.36) indicate a uniform structure and the complete absence of a brittle surface layer characteristic of traditional nitriding methods.

As can be seen from Table 3.10, the geometric dimensions of the control samples remain almost unchanged within the accuracy of 1–2 µm.

2. DIN 1.773 steel samples and control samples of the same steel were used as substrates.

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*µ=350...370. The nitrided layer has a uniform structure, without any fragile surface layer.

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

3. 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.

The appearance of the nitrided layer on 24CrMoV5-5 steel is shown in Fig. 3.37.

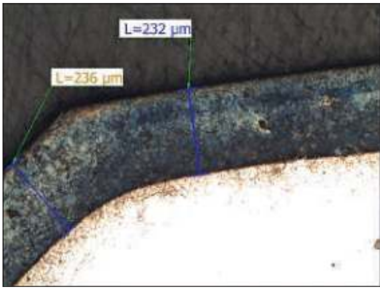


Fig. 3.37 The nitrided layer depth (×500)

Etched and unetched polished sections of the nitrided layer on 24CrMoV5-5 steel and hardness measuring points are shown in Fig. 3.38 and Fig. 3.39.

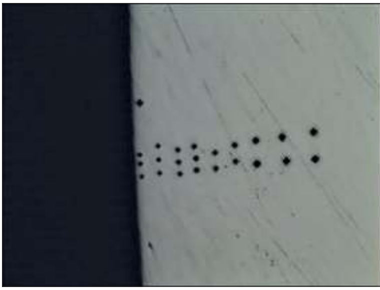


Fig. 3.38 Imprints of microhardness measurements (unetched polished section)

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.

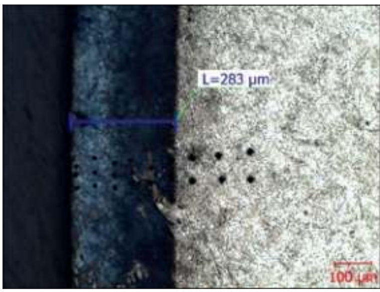


Fig. 3.39 Imprints of microhardness measurements (etched polished)

The study of the nitrided layer microstructure (Fig. 3.39) exhibit a uniform structure and the complete absence of a brittle surface layer, characteristic of traditional nitriding methods. 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.

4. 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 raised to 700 °C, the nitriding time was 4 hours. On the sample of the titanium alloy DIN 3.7165 (Fig. 3.40) the structure and thickness of the nitrided layer, the nitrogen content and the change in the microhardness were studied.

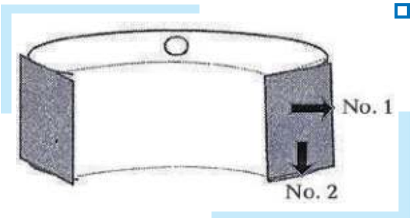


Fig. 3.40 Scheme of a technological sample from DIN 3.7165 titanium alloy

The microstructure of the sample outside the area of the nitrided layer is α solid Ti solution.

The measurement of the nitrided layer hardness is performed on unetched samples on the hardness tester 'Leco AMH 43' with a load of 50 g.

The magnitudes of change in the hardness of the nitrided layer, starting from the edge, are presented in Fig. 3.41, 3.42.

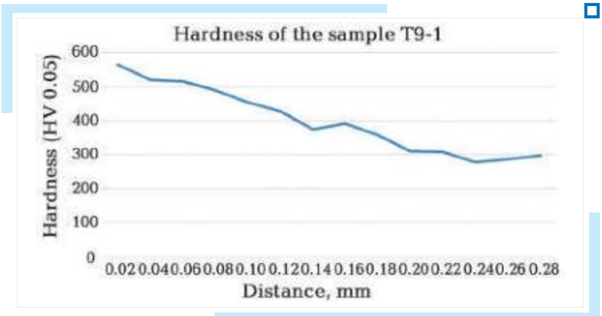


Fig. 3.41 The hardness of the nitrided layer. Side No. 1

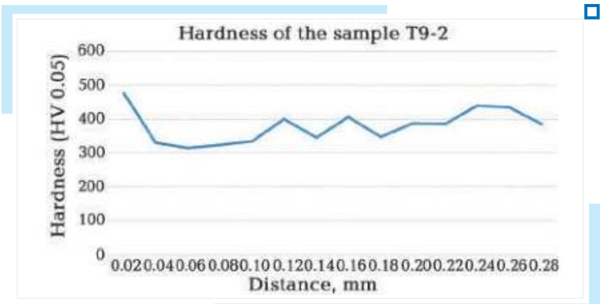


Fig. 3.42 The hardness of the nitrided layer. Side No. 2

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 microstructure and thickness of the nitrided layer of the sample from side No. 1 and No. 2 are shown in Fig. 3.43 and Fig. 3.44.

On the surface No. 1, the presence of an alpha layer 4–5 μm thick is observed (Fig. 3.45). Appearance of microhardness measurements imprints represented in Fig. 3.47, 3.48.

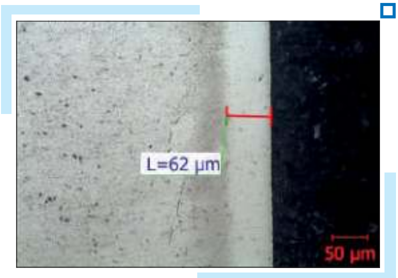


Fig. 3.43 The microstructure and thickness of the nitrided layer Side No. 1, ×200

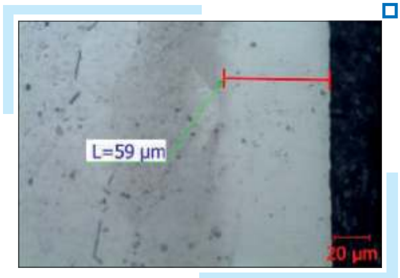


Fig. 3.44 The microstructure and thickness of the nitrided layer Side No. 2, ×500

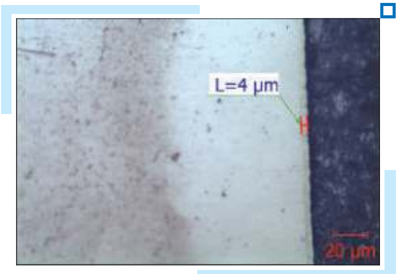


Fig. 3.45 Alphated nitrided layer from side No. 1, ×500

Fig. 3.46 Nitrided layer from side No. 2, ×500

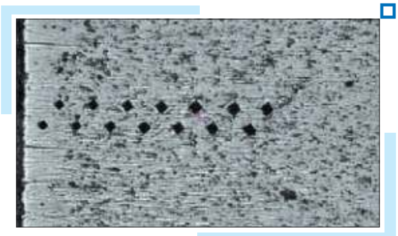


Fig. 3.47 The microhardness measurements imprints. Side No. 1

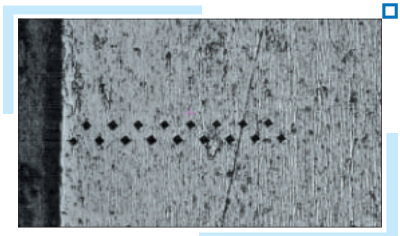


Fig. 3.48 The microhardness measurements imprints. Side No. 2

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.

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.

In case of Avinit N plasma nitriding the process of creating a nitrided layer is intensified by 3–5 times compared with the treatment with the traditional method of ion nitriding in glow discharge.

The hardness of the products is increased due to obtaining a uniformly strengthened nitrided layer without distortion of the parts while maintaining the original geometric dimensions.

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
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
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

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.

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 σ_{max} 1000 MPa.

Test results based on 1.000.000 cycles (rolling with slip with contact loads σ_{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.

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.

Examples of plasma nitriding Avinit N:

1. The gear is manufactured as per accuracy 4 degree (up to 1 μm) with Avinit plasma nitriding. The original geometric dimensions before and after nitriding are stored within the accuracy of 1–2 μm.

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

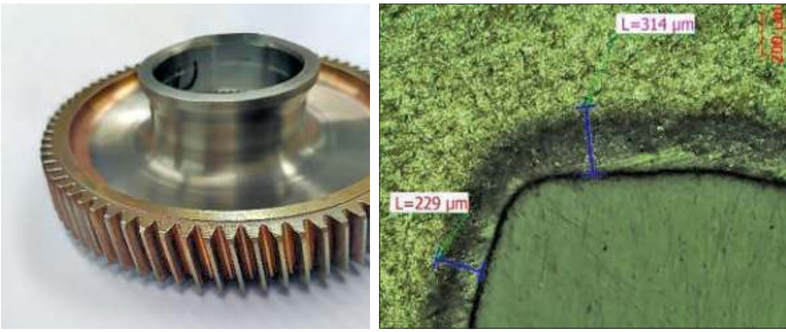


Fig. 3.49 Plasma nitriding of Avinit N steels 34NiCrMoV14-5. Depth of the nitrided layer 0.3 mm. Microhardness $H\mu$=730–930

2. Freewheel clutch separator. Steel 41CrAlMo7.

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.

Fig. 3.50 Freewheel clutch separator. Plasma nitriding of Avinit N+coating Avinit C

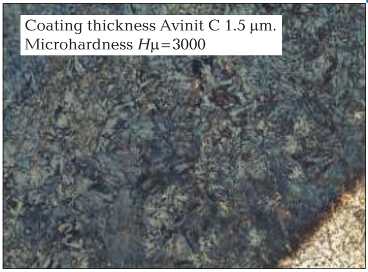


Fig. 3.51 The structure of the nitrided layer. Depth of the nitrided layer 0.2 mm. Microhardness $H\mu=730–830$

3. Plasma nitriding of parts from steel 24CrMoV5-5.

Plasma nitriding of Avinit N steels that are not typical nitrided steels provides the same, and often higher, properties of parts at lower manufacturing costs than for nitrided steels.

Fig. 3.52 Plasma nitriding of Avinit N

4. Plasma nitriding of parts from titanium alloy.

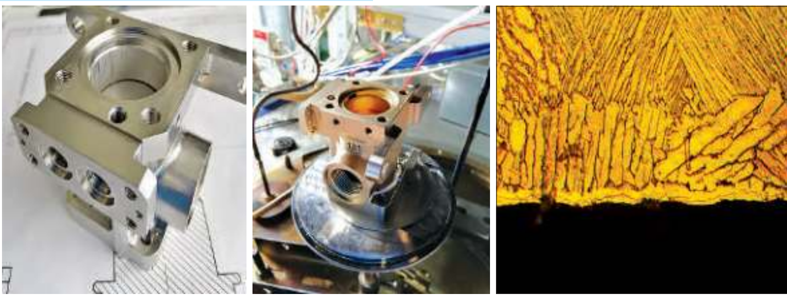


Fig. 3.53 Housing from DIN 3.7165 titanium alloy. The structure of the nitrided layer on the inner cylindrical part of the housing surface

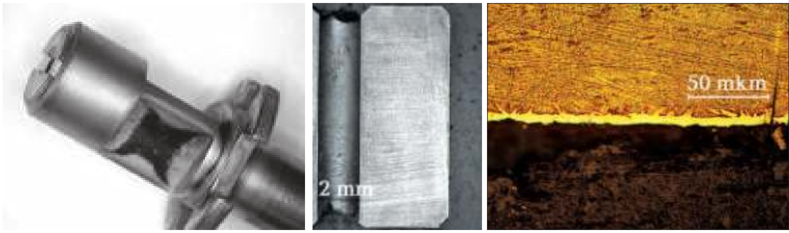


Fig. 3.54 Slide valve. Structure of nitrided layers on slide valve surface

Fig. 3.55 Authors process development

3.5 Antifriction coatings based on molybdenum disulfide with respect to unit and engine parts building

We have elaborated various processes of depositing MoS_2 coatings both by PVD (magnetron) and CVD methods.

The processes of MoS_2 deposition are low-temperature — the deposition temperature does not exceed 200 °C, which allows to deposit the coatings on precision, pre-heat treated surfaces, including aluminum alloys, and bronzes.

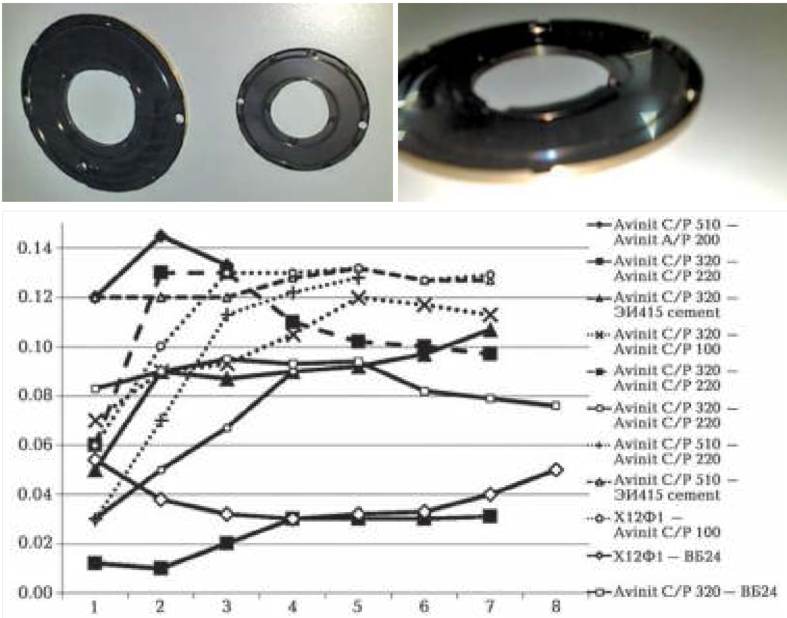
The use of the CVD method allows to implement the unique opportunities for applying disulfide coatings on the precision parts inner surfaces.

Processes of coating deposition from hardened molybdenum disulfide have been elaborated.

MoS_2 coatings can be used individually or in combination with PVD or CVD hard and super hard coatings [20].

The coatings thickness can usually range from 1 to 10 microns. Coatings can be extremely thin and ultrathin (0.1 µm and less), which is extremely important when coating is deposited on precision components

The studies of hardened molybdenum disulfide coatings were performed on a REM-106I scanning electron microscope with X-ray energy dispersive analysis system designed to measure the linear dimensions of topology and surface microrelief parameters of various objects in the hard phase and to measure the mass fraction of elements in the composition of objects using X-ray microanalysis.



❐ **Fig. 3.56** Parts of aircraft units coated with molybdenum disulfide

❐ **Fig. 3.57** Parts of aircraft units coated with molybdenum disulfide

The hardened Cu-MoS_2 coatings were deposited by magnetron sputtering of the combined annular and sectoral MoS_2 and Cu targets onto the steel substrates. The coatings thickness is approximately 1.8–3.0 microns.

The tribological studies performed indicate that in the conditions of dry friction, the obtained coatings have very low friction coefficients which can be compared with coatings of pure molybdenum disulfide, but possess significantly better wear characteristics.

3.6 Nanocomposite functional Avinit coatings in transport engineering

Hard and superhard coatings, which are multilayer multicomponent structures, combine both high hardness and wear resistance and low values of friction coefficient, which is important when used in friction tribo-couples to prevent adhesion or reduce damage to an acceptable level.

Given the excellent tribological properties that provide new vacuum-plasma surface treatments and functional coatings, it is advisable to use Avinit nanocomposite functional coatings in transport engineering, in particular, to improve the performance of diesel engines.

Coating activities on diesel components are conducted both for reducing wear and friction of parts using antifriction coatings, and for development of new multifunctional nanocomposite coatings using nanotechnologies with improved tribological properties for use in various tribo-couples:

a) for cylinder-piston group (CPG) parts:

- pistons;
- valve oil and compression piston rings;
- piston fingers, valves and sleeves.

b) for parts of crankshaft assembly (KSA):

- crankshaft;
- steel-aluminum and steel-bronze crankshaft bearing and connecting rod liners.

Tribological tests of Avinit coatings were carried out on samples of heat-resistant deformed AISI/ SAE A92618 alloy with sliding friction under boundary lubrication conditions [1, 58, 59]. The developed nanocomposite coatings on the AISI/SAE A92618 alloy of the corresponding compositions allow to prevent scoring when working in pair with the sleeve cast iron in the presence of friction-sliding at the boundary lubrication conditions corresponding to the working conditions of the cylinder-piston group parts of diesel engines. In this case, the relative increase in resistance reaches 20–80 times, and the wear of the counterbody decreases by 4–5 times.

Avinit C220 coatings are characterized by the best combination of wear resistance, durability with respect to the sleeve cast iron and the antifriction properties of the coatings studied.

Avinit C220 coatings as antiscoring wear-resistant coatings were deposited to pistons D80.0406-1 of deformed heat-resistant aluminum alloy AISI/SAE A92618 to carry out bench testing.

Table 3.12 The values of the friction coefficients f_{fr}, at various loads

No.	Coatings	The ether value f_{fr} at load, P, kN									Notes
		0.2	0.4	0.6	0.8	1.0	1.2	1.4	1.6	2.0	
1	–	0.03	0.01	0.01	0.01	0.01	0.03	–	–	–	
2	Avinit C140	0.06	0.09	0.12	0.12	0.12	0.12	0.11	0.115	0.113	
3	Avinit C120	0.17	0.15	0.14	0.14	0.13	0.14	0.13	0.135	0.137	
4	Avinit C120	0.09	0.1	0.12	0.12	0.12	0.12	0.12	0.126	0.13	
5	Avinit C150	0.016	0.02	0.07	0.11	0.11	0.11	0.11	0.11	0.11	
6	Avinit C320	0.03	0.07	0.12	0.12	0.12	0.12	0.12	0.12	0.116	
7	Avinit C220	0.022	0.04	0.06	0.11	0.11	0.1	0.11	0.11	0.116	I load
8	-'-'-	0.022	0.005	0.005	0.006	0.007	0.007	0.008	0.005	0.008	II load

Bench tests of the field pistons made of deformed heat-resistant aluminum alloy AISI/ SAE A92618 with developed antiscoring wear-resistant coatings Avinit C220 for D80 type diesels were carried out as part of a single-cylinder diesel engine with maximum approximation to field operating conditions.

The developed Avinit C220 antiscoring coatings for D80 type diesel engines have been fully factory-certified. Diesel engine has passed the running and acceptance tests.

Test results confirmed the high efficiency of the coatings developed.

Fig. 3.58 Pistons D80.0406-1 of the D80 diesel engine with Avinit C220 anti-scoring wear-resistant coatings

In the conditions of piston rings operation comparative tribological study of the developed coatings and coatings from electrolytic chromium used in serial production were carried out.

Studies were performed to determine tribological characteristics in friction couples with sleeve cast iron indicated that the greatest stability have Avinit C310 nanocomposite coatings.

Table 3.13 Comparative tribological study of piston rings with the developed coatings

Coating material	Value f_{fr}	Microhardness, HV_{50}, kgf/mm^2	
		Initial	The friction path
Avinit C310	0.029	1503	1720
Avinit C220	0.032	1507	2335
Avinit C240	0.031	2022	2212
Chrome ring	0.101	1057	755

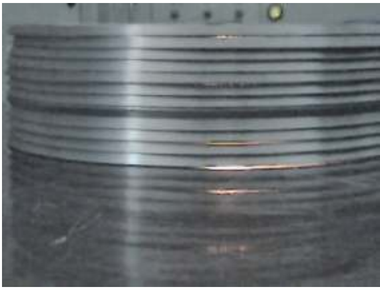


Fig. 3.59 Valve oil piston rings with Avinit hardening coatings

The test results of the coatings studied for the determining the friction coefficients (f_{fr}) show that the use of piston rings with composite coatings provides higher wear resistance and better friction characteristics sleeve — piston ring compared to the used electrolytic chrome coating [60, 61].

These studies have provided the basis for the development of hardening coating technologies on valve oil piston rings of diesel locomotive diesel engines.

The developed Avinit hardening nanoproducts for diesel engines compression rings provide increased nominal power output, reduced specific fuel consumption by reducing the friction loss of the compression rings while reducing the total number of piston rings.

The use of Avinit multifunctional composite coatings is also effective for increasing the performance of crankshaft group (CSG) of diesel engines.

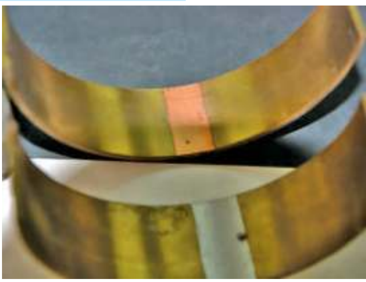


Fig. 3.60 Avinit anti-friction coatings on the sliding bearings (liners) work surface for internal combustion engines

Fig. 3.61 Technological equipment for producing liners with Avinit hardening anti-friction coating

For steel-bronze and aluminum alloy liners, Avinit C610 running-in coating based on AlCuSn and Avinit C710 quasi-crystalline coating based on AlCuFe have been developed.

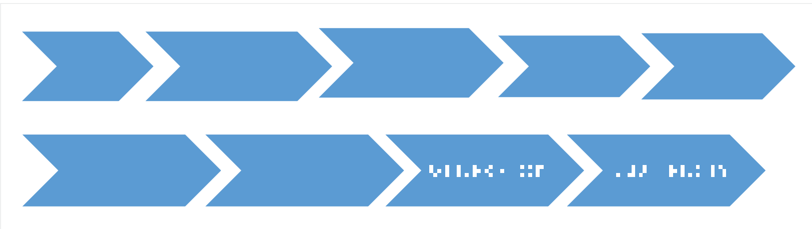
The works performed to increase the service life of diesel engines by expanding the scope of possible use of new generation coatings can serve as a basis for creating a 'repair-free diesel engine' that is competitive in the world market.

Fig. 3.62 Diesel unit

3.7 Application of ion-plasma methods for the production of thin-film solid oxide fuel cells (SOFC)

JSC FED is actively working for application of ion-plasma methods aimed at production of thin-film solid oxide fuel cells (SOFC).

Obtaining electric energy by traditional methods is provided by converting chemical energy into electric energy through thermal and mechanical energy.



Due to significant energy losses caused by the conversion of heat into mechanical energy, the efficiency of thermal power plants equals to a maximum of 45 %, diesel units — 30 % and gasoline units — 20 %.

In the case of direct conversion of chemical energy into electrical energy using electrochemical fuel cells: efficiency can in principle reach about 100 %.

Therefore, this method refers to 'highly efficient methods of energy conversion [62—64].

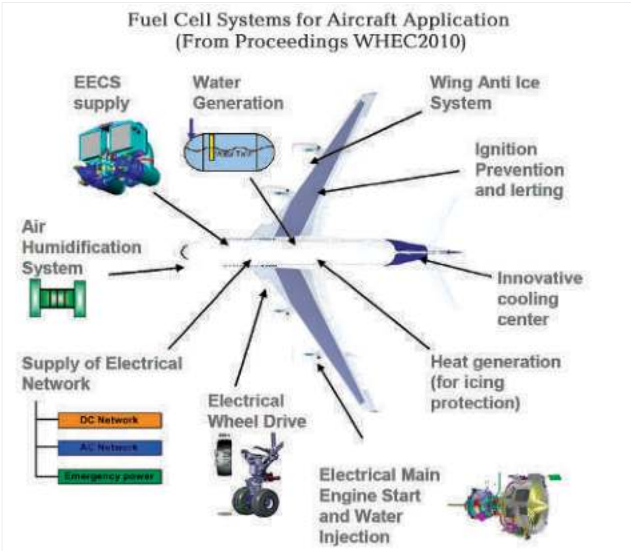


Fig. 3.63 Multifunctional fuel cell system

Thin-film version of the fuel cell. The most promising direction for increasing the efficiency of fuel cells is to reduce the thickness of the components of the PEN structure while simultaneously optimizing the structural characteristics [65–68].

Thin-film versions of solid oxide fuel cells, of course, will have a decisive place in future serial designs of electrochemical generators. The obvious advantages of thin film — reducing the internal resistance of the source and compaction of the structure — despite the difficulties of implementation.

Vacuum-plasma methods of forming a composition for film thermoelements (electrolyte — electrode — current collection) have the widest possibilities for creating appropriate materials with the necessary set of properties.

The most acceptable method for application of thin films of electrode and electrolyte multicomponent oxide materials is electron-beam plasma method (AIS).

Fig. 3.64 Layer of layers of single FC with electrolyte based on barium cerates

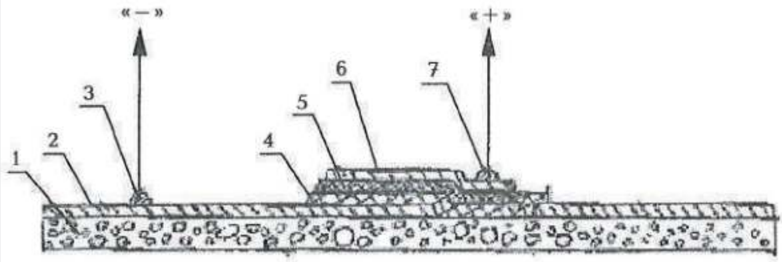


Fig. 3.65 Sketch of a single fuel cell: 1 — Bearing base; 2 — Collector; 3, 7 — Current leads; 4 — Cathode; 5 — Solid electrolyte; 6 — Anode

Physical principles laid down as the method basis, allow to more fully resolve the issue of coating with a specific chemical and phase composition (and as a special case — reproduction of a spattering target's composition), and to ensure the necessary density or porosity of the layer applied.

The application of vacuum-plasma methods to the problem of creating materials for thin-film SOFC allows to assure easily reached and controlled conditions for synthesis of deposited chemical compositions and mechanical strength of the coating.

Using the method of atomic-ion plasma sputtering (AIS), technological procedures have been developed for producing thin gas-tight VSN electrolyte coatings and porous electrode (anodic and cathodic) layers for the manufacture of experimental SOFC elements [69].

Studies performed on BCN samples indicate good reproducibility of the chemical and phase composition of the target in the $BaCeNdO_3$ coating.

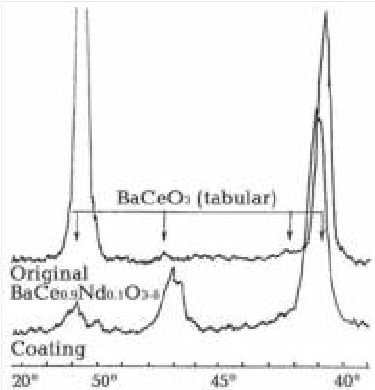


Fig. 3.66 Comparative diffraction spectra of BCN in coatings and spray targets

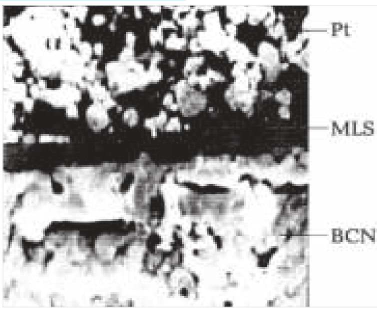


Fig. 3.67 Raster microscopy of the test specimen: BCN (base) — MLS (coating) — Pt (baked paste), ×3300

The study of fractures of samples with electrode coatings using raster microscopy indicates good adhesion between the backbone of BCN and the applied coatings.

Technological procedures have been developed for the production of collector nickel and nickel-chromium coatings for the experimental elements SOFC based on the BCN electrolyte using the vacuum arc spraying method providing current-collecting coatings with a given level of thickness, composition, adhesion, microhardness and adopted as a baseline the creation of film compositions SOFC [69–71].

Table 3.14 Characteristics of current collector coatings

No.	Material of fuel current collecting device	Width of current collecting element, mm	Thickness, μm	Number
1	Nickel	2	6	2
2	-'-'-	4	3	2
3	-'-'-	6	2	2
4	Nickel+Chromium	2	6	2
5	-'-'-	4	3	2
6	--'-'-	6	2	2

The thickness of the collector coatings for the nickel collector is 3.8–4.2 μm, for nickel-chromium (4.3–4.7) μm, the microhardness for nickel coatings is 1450 MN/m^2, and for nickel-chromium coatings 4550 MN/m^2.

Experiments have been investigated and experimental technologies have been developed for deposition of thin layers of the BCN electrolyte, Ni+BCN anodes, and collectors of Ni and Ni+Cr using vacuum-plasma methods [69–71].

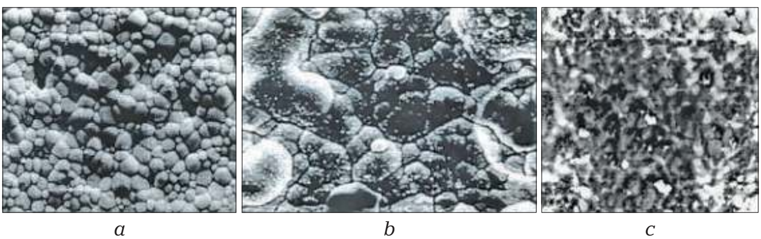


Fig. 3.68 Surface morphology: *a* — BCN electrolyte; *b* — Ni-BCN layer; *c* — Ni-Cr coating at Ni-BCN anode, ×1500

Bench tests were carried out using the technologies of thin-film OPE batches [72].

In the temperature range (600–900) °C, the electrochemical characteristics of thin-film single fuel cell with the BCN electrolyte (BCN electrolyte working area — 3.14 cm^2) with a thickness of (1.7–1.9) mm, made by pressing and subsequent sintering, and electrode layers of Ni-BCN (11.4 mg/cm^2), MLS (1.1 mg/cm^2), made by the methods of atomic-ion plasma spraying and electric arc spraying, as well as with additional platinum collector.

Table 3.15 Electrical characteristics of a thin film unit fuel cell

Test temperature, °C	Unit fuel cell Ni-BCN/BCN/MLS + Pt		
	U, B	I, mA/cm^2	P_{max}, mW/cm^2
600	0.95	0	9.5
	0.593	14.1	
	0.455	20.0	
	0.21	31.2	
700	0.9	0	15.3
	0.677	16.5	
	0.312	44.7	
800	0.842	0	21.1
	0.7	16.5	
	0.584	30.3	
	0.39	52.9	
900	0.81	0	26.7
	0.71	16.5	
	0.61	32.9	
	0.45	58.8	
The maximum power taken from the thin film unit fuel cell in the temperature range (600–900) °C is 27 mW/cm^2			

The level of developments reached by FED, JSC in respect of composition materials while using ion-plasma and plasma-chemical methods creates pre-requisites for changing properties with respect to traditional materials by 2–3 and more orders, reducing operating temperature to 400–600 °C and other, which would allow to develop revolutionary designs of thin-film FC (with thickness by 20–30 times less than tubular option) and serial production technologies for their manufacture having the following areas:

- application of thin-film composition on the developed structure taking with view to technological limitations for deposition methods;
- production of composite materials consisting of layers: gasproof electrolyte and electrode layers with thin-film current collection contacts;
- ensuring the separation of gas mixtures with a ceramic electrolyte layer with a thickness <20 μm;
- minimize the thickness of the film electrolyte and other functional layers of the fuel cell.

Increasing the adhesion strength of the layers and the corrosion resistance of the collector contacts and electrode layers in working media to ensure the efficiency of the structure during the entire operation process.

4

Introduction of developed Avinit technologies into serial production

4.1 Industrial technologies for deposition of hard and super-hard Avinit nanolayers

JSC FED possess 26 legally effective patents. The quality management system is certified by 'Bureau Veritas Quality International' in accordance with ISO-9001, ISO-14001, ISO 9100:2009.

Fig. 4.1 Certificates of JSC FED

On the basis of extensive research and experimental studies carried out for the choice of nanocoatings materials for the manufacture of friction couples, the development of the optimum composition of coatings and technologies for their obtaining for the developed parts in pumps and regulators of units of fuel supply and control of aircraft engines, it was experimentally substantiated that introduction of nanostructural and nanolayer materials in serial production of hydraulic units provides high reliability of work of serial and new designs of units and increases adjustment and overhaul period by 5–20 times.

Unique solutions are offered to provide higher reliability and extended service life of various critical aircraft components in unit- and engine building based on surface modification technologies and Avinit functional coatings deposition.

More than 30 advanced technologies for deposition of nanolayer low-temperature (≤150–200 °C) coatings to increase the reliability and service life of friction couples in unit building, have been introduced into serial production.

The use of slide valves coated with Avinit C 320 in hydraulic units increases their service life from 50 to 4.000 hours (!).

Fig. 4.2 Parts of Avinit C310 serial aviation hydraulic units with nanocoating

Fig. 4.3 Hydraulic units cradles with Avinit C310 nanocoating

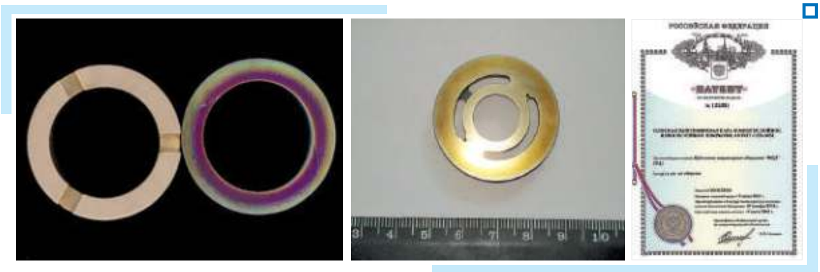


Fig. 4.4 Slide valves of hydraulic units coated with Avinit C320 [21–24]

Fig. 4.5 Slide valves of hydraulic units with Avinit C 310 coating [25–28]

The service life of units with slide valves coated with Avinit C 310 increases from 200 to 2.000 hours.

4.2 Avinit N Series Plasma Precision Nitriding

Avinit N plasma precision nitriding is already broadly used in serial production JSC FED instead of the traditional methods of ion nitriding, liquid and gas nitriding.

Advantages of Avinit N Plasma Precision Nitriding. 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;
- 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.

Plasma precision nitriding processes eliminate the disadvantages of traditional industrial nitriding processes (traditional ion nitriding, liquid and gas nitriding), increase the performance of parts, expand the range of materials to be processed.

Fig. 4.6 Serial parts of aviation hydraulic units, nitrided by plasma precision nitriding

Fig. 4.7 Serial nitriding of gear wheels (34NiCrMoV14-5)

Fig. 4.8 Serial nitriding of titanium DIN 3.7165 housings

4.3 Industrial technologies of thermal-vacuum treatment and diffusion welding

Available in JSC FED a stock of thermal equipment of both own manufacture (design, manufacture, launching and debugging, elaborating the modes and techniques of chemical-thermal treatment that provide the desired properties of the product with a hardened surface, the development of industrial technologies), and the best foreign and domestic manufacturers can fully meet the needs of serial production in accordance with the technologies of vacuum heat treatment, chemical-thermal treatment (plasma nitriding, cementation and nitro-cementation) and diffusion thermal and electrical arc welding.

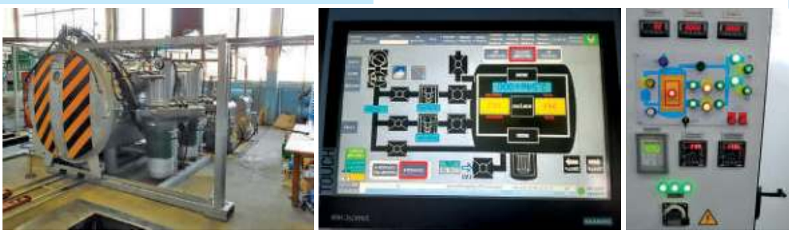


Fig. 4.10 Upgraded automated vacuum-thermal installation for diffusion welding and thermal treatment with horizontal loading, forced gas cooling of the melting charge

Fig. 4.9 One-chamber vacuum automated installation of diffusion welding and heat treatment of parts

One-chamber vacuum automated installation of diffusion welding and heat treatment of parts with forced gas cooling of melting charge is designed for diffusion welding and heat treatment of machine parts in vacuum (annealing, tempering, normalization, hardening of alloy steels and other heat treatment processes).

Modern measurement and control systems based on the industrial serial controller SIEMENS can significantly increase the reliability of the installation, fully program the entire technological process of heat treatment and conduct it in automatic mode.

The equipment is widely used in batch production for thermal and chemical thermal treatment of various industrial parts.

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