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RELIABILITY, STRENGTH, WEAR RESISTANCE  
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## Wear Resistance of Composite Plasma Coatings with Graphite

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**Abstract**—The wear-resistant composite solid lubricant coatings formed by atmosphere plasma spraying method of cladding Ni[Cg] powder in relation to the extreme working conditions of friction units of aircraft engines and power plants have been investigated. Examples of studies of technological processes of the plasma spraying of protective coatings on elements of liquid-propellant rocket and gas turbine engines have been given, and the possibilities of sealing technologies to increase their service life have been shown. The evaluation of efficiency of coatings obtained by plasma spraying from mechanical mixtures of powders based on Ni[Cg] in relation to power plants of vehicles based on free-piston engines.

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It is known that composite materials obtained by plasma spraying from the cladding by nickel powders of graphite, boron nitride BN, and calcium fluoride CaF<sub>2</sub> are effectively used for extreme operating conditions of friction units, seals, and structural elements of modern aircraft engines and power plants for vehicles. A comparative analysis of these materials used as the solid lubricant coatings [1] has shown that graphite–metal mixtures with a volume content of graphite within 10–35% obtained by plasma spraying from cladding Ni[Cg] powder are the most effective.

The plasma coatings based on cladding nickel–graphite powder Ni[Cg] traditionally belong to the class of materials that are worn out and abradable, but resistant to erosion, oxidation, and temperature. They are used to line the inserts of the gas turbine engine stator, as well as abraded and run-in during the beginning of blade rotation, as well as sprayed on the construction elements of labyrinth seals of various types in order to ensure the minimum radial and/or axial clearance. The use of these coatings significantly reduces the leakage of air and working gas through clearances and increases the engine efficiency, but also greatly simplifies the assembly process and facilitates the repair and reinstatement of inserts and labyrinth seals.

However, the use of coatings based on Ni[Cg] as antifriction coatings, which ensures the designed wear resistance in friction units, such as the sealing rings of pumps, the rotary sleeves of blades, combustion engine cylinders, and other similar friction pairs, is extremely limited. In these cases, this means that antifriction coatings based on Ni[Cg] that work with minimal coefficients of friction prevent the seizure of metals and have lubricating ability with the minimum possible wear.

In this article, the results of a study of the coatings based on Ni[Cg] powder have been proposed for use as wear-resistant solid lubricant coatings in manufacturing and repair technologies to ensure the protection of constructive units of products working under wear conditions in a wide range of temperatures and pressures. Ni[Cg] powder belongs to the class of composite cladding powders (or powders with coating). The initial materials of powder particles are as follows: the core is graphite and the cover is nickel Ni. Three types of domestic Ni[Cg] powders are known: PNG-85, PNG-80, and PNG-75 containing 85 ± 2%, 80 ± 2%, and 75 ± 2% of nickel, respectively, while the rest is graphite. The powder particle size is in the range of 40–160 μm, the powder fluidity is in the range of 60–100 s, and the apparent density is in the range of 1.5–3.5 g cm<sup>-3</sup>. Ni[Cg] powders manufactured by Western companies: Metco–Sulzer, Sherit–Gordon, Shtark, etc. have approximately the same characteristics. Cladding graphite with nickel is carried out mainly by one of the following methods: gas-phase method (cladding from carbonyl gas phase in the vibration-boiling layer) or the chemical method (chemical nickeling by the method of Ni reduction from aqueous solutions).

One of the first developments of wear-resistant composite coating with graphite based on Ni[Cg] was related to the need to raise the tribotechnical properties of the inner surface (with the diameters of Ø43H9, Ø70H8, and Ø81H8, respectively) of the floating rings of the seal of the cryogenic pump of liquid rocket engine made of Br010 bronze (10% tin, the rest is copper), working together with counterpart of anodized aluminum. The coating played the role of solid lubricant under fairly hard operating conditions.

At the point of time of starting and stopping this pump, extreme shearing forces (tangential to the inner surface of sealing ring) arose, the main reason for which is dry friction, which occurs under the action of dead weight of the ring and angular momentum at the time of starting and stopping. This led to the wear of interfacial parts and their hot-spot oxidation (burns), since at impulse contact, the dry friction was accompanied by an local leap in temperature ( $\geq 600^\circ\text{C}$ ). The orientation of the microrelief, which coincides with the direction of the rotation of the parts, is unprofitable with respect to antifriction properties because of the increase in the actual field of contact. The presence of aluminum oxides in addition to copper oxides in wear products is evidence of the abrasive nature of the beginning of wear. The particles of oxide film shearing under dry friction under extreme starting conditions extracted the material from the surfaces of contacting parts, while these particles were introduced to the surface and detained friction products on themselves, forming galling stringers.

The operating conditions were defined in the following terms: a counterpart of anodized aluminum with a heightened microhardness of up to 400 HV, a working temperature range of friction pairs is of 213–1073 K, and thermal cycling (at least 100 cycles) in this temperature range. To protect the work surfaces of the sealing rings, nickel–graphite antifriction coatings were deposited on their inner diameter by plasma spraying. The initial material for spraying was PNG-80 powder; cladding by nickel of graphite was carried out from the carbonyl gas phase in the vibration boiling layer.

The plasma sprayed coatings had natural porosity, the presence of which in this case played a positive role in ensuring the anti-friction properties, first, because the contact discreteness of these layers is more than on solids and, second, their surface served by peculiar reservoir to collect and retain the lubricant and the wear products. The contact discreteness also decreased the possibility of formation of seizure bridges. The heterophase structure of [Cg]Ni coatings, in which nickel is the matrix and graphite is the filler, provided high antifriction properties and the ability to safe-lubrication due to the formation and reinstatement of thin oriented film in the friction zone.

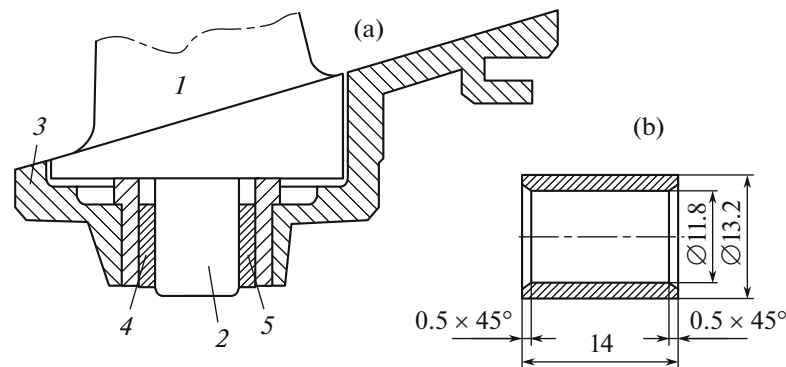
The main feature of this solid lubricating coating is that it is a composite material in which nickel is the metallic bond (matrix), the pores of which contain graphite particles. The nickel matrix defines the fastening method of the solid particles of graphite lubricant in the compositional material, prevents the lubricant particles from being removed from the contact area, and takes the maximum carrying load of the composite material of coatings. The developed solid lubricant coating (SLC) technology was recommended and used to produce a real product for protecting the inner surfaces of floating sealing rings of cryogenic turbine-pump assembly and the gas generator of the liquid rocket engine [2].

The bench tests of floating rings with Ni[Cg] coatings were conducted that consist of a technological turbine-pump assembly of the liquid rocket engine under the following conditions: the working medium is liquid oxide, the loading cycles is  $\geq 100$  (running time is  $> 5$  h) according to the technical specifications, the shaft rotation speed is  $26 \times 10^3$  rpm and the gap between the shaft and the coating surface is within 0.15–0.18 mm. These conditions completely simulated the natural conditions.

The conducted tests revealed that the coating deposited on the floating sealing rings, eliminated the appearance of defects on the contacting surfaces, and completely removed the limitations on the working time and to the number of engine starts.

Further study of the spraying technology and properties of plasma-sprayed SLC based on nickel graphite Ni[Cg] were carried out on samples of INCO718 steel (the domestic analogue is KhN60Yu steel) intended for use in the friction pairs of the bearing unit (contact of shaft-sleeve type)–rotary stator blade of compressor, in which the samples of INCO718 steel served as the counterpart. The operating conditions of the friction pairs are as follows: the temperature is in the range of 20–400°C, the contact pressure is 80 MPa, and the environment is air. Figure 1 presents the constructive arrangement of the rotary friction unit.

The reciprocating rotary (reversible) cyclic friction pair is used in the considered friction unit. Under the condition of validity, in the actual engine design, it is possible to use only the version with movable shaft (pivot 2) with a sleeve with coating pressed on it (Fig. 1a). The force that loads a hinge is the resulting of aerodynamic forces attached to the blade profile of the guide vane, the direction vector of which when turning the blade also rotates. In this case, this type of sliding bearing loading, in which the area of max-



**Fig. 1.** Friction unit of rotary blades of gas turbine engine compressor: (a) unit construction; (b) sleeve with coating; (1) stator rotary blade, (2) rotary pivot, (3) carter frame, (4) intermediate sleeve, (5) sleeve with coating on  $\varnothing 13.2$  mm.

imum contact pressures moves on the sleeve surface within the specified angle of rotation ( $\pm 12^\circ$ ), is implemented.

Using sleeves with coating in this friction unit simplifies the dimensional processing of the engine carter frame, creates an opportunity to use various materials when creating the friction pair without changing the fabrication method of the frame and makes it possible to use a wide range of methods to modify the surface friction in those sleeves. Furthermore, the known limitations that take place in the case when the bearing holes made directly in the frame decrease and repair the ability of the friction pair increases.

Since an important characteristic of the coatings is the adhesion to the substrate, an evaluation of the influence of surface preparation prior to the coating was made. The chemical composition of the surface of the samples was determined not only on the coating surface, but also before its deposition on the sample surface before spraying. For this electron GLAM-100 Auger-spectrometer of Vacuum Generation Company was used, which has been installed in the UHV chamber of Varian brand. The purpose of experiments by determining the composition of the surfaces of samples without the coating was to evaluate the influence of sand blasting and the queue time of the samples in air after this processing on the chemical composition and the surface quality of the steel sample before spraying the coating. The liquid blasting of the samples were fulfilled with fused alumina with a grain size of 80–100  $\mu\text{m}$ , at air pressure of 4–6 atm, at a distance from the nozzle exit section to the surface of 70–100 mm, and the nozzle axis inclination to the surface under treatment of 70°–90°.

After sand blasting, the samples were held in air for a specified time and placed in acetone for storage until they were installed in the vacuum chamber of the Auger spectrometer. After first series of the Auger spectra were obtained, the samples were processed by an argon beam with an ion energy of 5 keV for 5 and 20 min. The concentrations of chemical elements on the sample surface were calculated according to the data of the Auger spectra.

According to the measurement results, it was stated that grit blasting taking into account the queue time increases the concentration of carbon and oxygen on the surface of samples. During the further processing of samples by  $\text{Ar}^+$  ions, the surface continues to clear and the concentrations of oxygen and carbon decrease. Furthermore, after ion beam processing, the surface quality (roughness) hardly depends on sand preblasting. Then, NiCr layer was deposited on the treated surface of steel samples by plasma spraying method, which served as a sublayer to ensure the adhesion of the coating to the substrate; then, the Ni[Cg] working layer was deposited.

The surface layer composition of the sample with Ni[Cg] coating were studied using GLAM-100 Auger spectrometer too. The analysis of spectra that determine the chemical composition of coating surface and at a depth of 5000  $\text{\AA}$  revealed that the main coating components are carbon, nickel, and oxygen, which are distributed evenly in depth. The processing parameters of the technological process of forming Ni[Cg] coating on INCO718 steel, as well as its subsequent sealing are presented in Table 1.

The best results on coating compaction were obtained using the hot isostatic pressing in the gasostatic extruder of the Crucible brand, with the following maximum values of operating mode parameters: the temperature is 970°C and the argon pressure is 160 MPa. The pictures (Fig. 2) of the surface of deposited Ni[Cg] coating before and after processing in the gasostatic extruder were obtained using scanning elec-

**Table 1**

No.	Name of the processing operation	Parameters and conditions of processing	Parameters of surface and coating layer, commentary
1	Sand blasting the coating surface	Al <sub>2</sub> O <sub>3</sub> powder, 63 μm; air pressure, 0.4 MPa; processing distance, 150 mm	Roughness $R_a = 0.7-1.1 \mu\text{m}$
2	Plasma spraying of Ni–Cr sublayer	Ni–Cr powder, 50–100 μm; current, 400 A; voltage, 40 V; volume-flow of plasma-forming gases: argon, 33.7 L/min; nitrogen, 10.0 L/min; volume-flow of carrier gas: nitrogen, 1.2 L/min, spraying distance, 100 mm	Powder is fed into the channel of RP-6 plasma-tron nozzle; sublayer thickness is 0.65–0.1 mm
3	Plasma spraying of [Cg]Ni layer	[Cg]Ni powder, 50–100 μm; current, 400 A; voltage, 40 V; volume-flow of plasma forming gases: argon, 33.7 L/min; nitrogen, 7.5 L/min; volume-flow of carrier gas: nitrogen, 1.4 L/min; spraying distance is 100 mm	Powder is fed into the channel of RP-6 plasma-tron nozzle; sublayer thickness 0.6–0.9 mm
4	[Cg]Ni coating compacting: Cold compacting  Hot gasostatic pressing	HP-500 hydraulic press, environment is air; room temperature, pressing pressure up to 350 MPa  Gasostatic extruder of Crucible brand, environment is argon; temperature within 920–960°C; pressure within 120–160 MPa	
5	Mechanical treatment of coatings	Speed 250 rpm; feed 0.1–0.2 mm/rev.	

tron microscopy (SEM) in a Camscan electron microscope. It can be seen that processing in gasostatic extruder significantly changed the nature and quality of Ni[Cg] surface of coating, the cracks and porosity disappeared and the hardness increased significantly (in order) from 0.6–1.2 to 6–12 GPa.

The preliminary tribotechnical tests of the obtained samples with coating were performed at the SMTs-2 friction machine by the ring–ring scheme (friction pair is Ni[Cg] – INCO718 steel) at a speed of  $V = 1$  m/s and contact pressure of  $P = 1$  MPa. An analysis of the dependences of the coefficient of friction  $f$  and wear magnitude defined by the weight method,  $I$  g/(m kg) (Table 2) revealed that compacting in a gasostatic extruder makes it possible to reduce the wear by almost three times compared with the noncompact Ni[Cg] coating. These tests revealed that the cold compaction of coatings can lead to an increase in the wear, which we believe is due to the fact that, at high compacting pressure of the coating (up to 350 MPa), the nickel frame is destroyed. Therefore, it is necessary to optimize the amount of coating pro-

**Table 2**

Processing kind	Mass wear, $I$ , g/(m kg)	Friction coefficient $f$
Without processing	$2 \times 10^{-6}$	0.1–0.2
HP-500 hydraulic press, draught pressure 350 MPa	$3 \times 10^{-6}$	0.1–0.2
Gasostatic extruder of Crucible brand	$5.5 \times 10^{-7}$	0.2

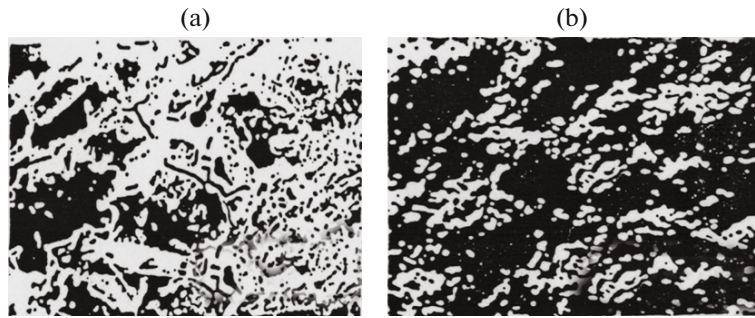


Fig. 2. Ni[Cg] surface before and after of hot isostatic pressing: (a)  $H_{\mu} = 0.6\text{--}1.2$  GPa; (b)  $H_{\mu} = 6\text{--}1.2$  GPa.

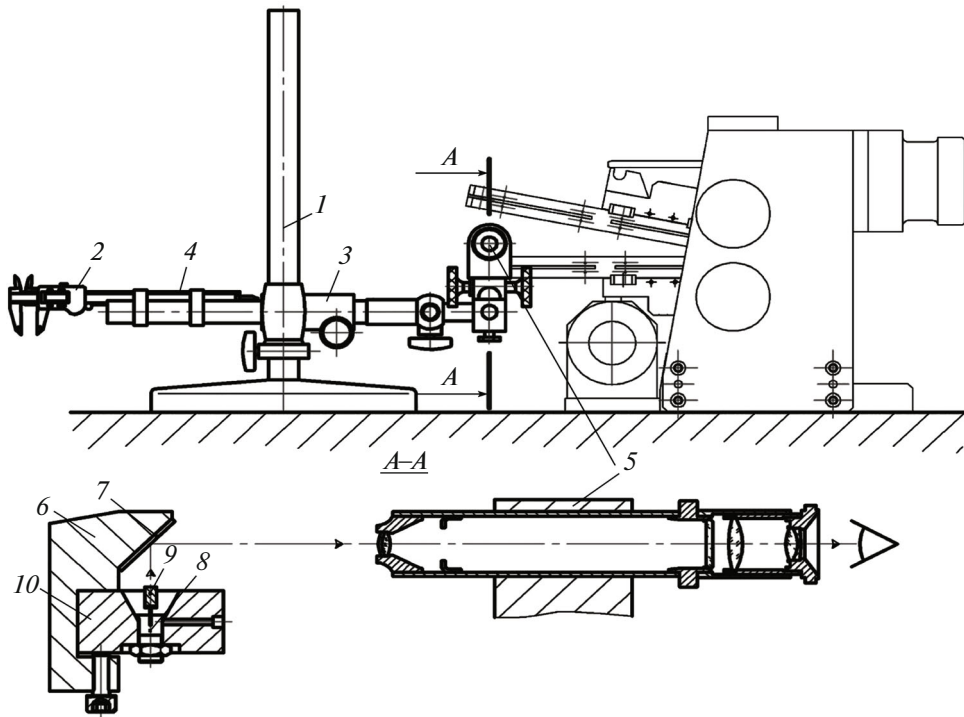
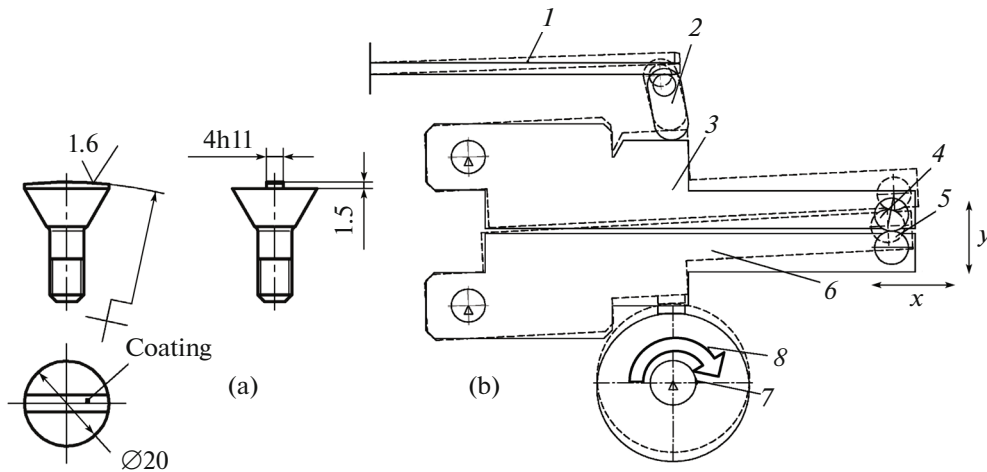


Fig. 3. Installation diagram of measurement of wear samples: (1) holder for measurement tools fastening; (2) digital slide gage, (3) fixed sleeve, (4) movable axis, (5) optical tube, (6) bracket, (7) mirror, (8) collet, (9) sample, (10) friction machine lever.

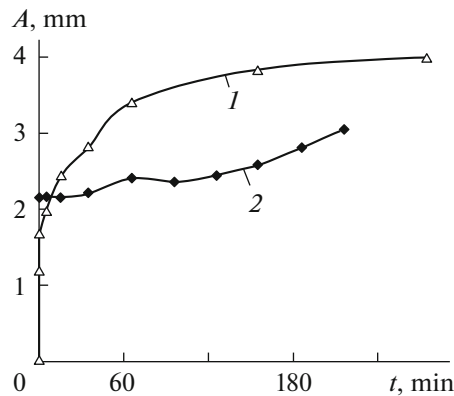
cessing pressure in the case of cold compaction. In all tests, the coefficient of friction was at approximately the same level of  $\sim 0.2$ .

A special study was devoted to the analysis of behavior of Ni[Cg] coating on INCO718 steel treated by hot isostatic pressing, by thick of  $\sim 150$   $\mu\text{m}$  in the end friction pair under the tests at UMT-1 friction machine with gradual increase of contact pressure up to 80 MPa and speed up to 3 m/s. It was stated that the coating withstands the desired service life of 100 min and, with an increase in the contact pressure from the initial amount of 27.5 to 80 MPa, the coefficient of friction decreases from 0.3–0.4 (during compression and running-in) to an amount of  $\leq 0.1$ .

After running for a service life of 100 min, the morphology of friction surfaces was studied by the scanning electron microscopy method in a Camscan electron microscope. It was determined that the wear of the Ni[Cg] coating + INCO718 steel friction pair at the end of the desired service life is accompanied by adhesion and the transfer of the part of coating material on the counterbody surface that led to its break-away and the formation of wear products. This was accompanied by the appearance of seizure effects, crack formation, and the oxidation of wear products. Therefore, further study of coating formation based



**Fig. 4.** Samples and kinematic scheme of their mutual displacement in friction machine: (a) sample drawing; (b) kinematics of samples displacement; (1) flat elastic element, (2) loading unit, (3) upper lever, (4) counterbody, (5) sample with SLC, (6) lower lever, (7) eccentric, (8) bearing.



**Fig. 5.**

on [Cg]Ni should be conducted in the direction of increasing their wear resistance while maintaining anti-frictionality (coefficient of friction).

Additionally, to evaluate the tribotechnical opportunities of materials used for plasma spraying, Ni[Cg] coatings of graphite powders, and cladding by different technologies, including the technology of Metco Corporation (electrolytic cladding) and the technology of domestic OOO Gipronickel Institute (cladding by deposition from gas phase) were compared. Figure 3 shows an installation diagram, which has been used on the results of the direct (without sample removal) measurement of wear chords and comparative tests that simulate the working conditions of stator blade rotation unit. Figure 4 presents the drawing of the samples under test and kinematic scheme of their relative motion.

It follows from Fig. 4b that fastened between two columns of friction machines (Fig. 3), two levers, i.e., upper lever 3 and lower lever 6, due to the rotation of bearing 8 fastened with eccentric 7 that provide the reciprocating rotary (within  $\pm 12^\circ$ ) motion of the segmental surfaces (Fig. 4a) of samples 4 (counterbody) and 5 (sample with the coating). Replaceable flat sheet spring 1 through unit 2 ensures the constant contact of samples 4 and 5 and their loading by pressure up to 80 MPa or more. The outer bearing race is in constant contact with lower lever 6.

The obtained results made it possible to state that the coatings using graphite powder cladding with nickel by deposition from the gas phase method have increased the wear resistance (by up to 1.5 times). The following can be seen from the dependence of wear chord  $A$  of the coating on time  $t$  shown in Fig 5: 1—Ni[Cg] of Metco corporation and 2—Ni[Cg] of OOO Gipronickel Institute.

**Table 3**

Composition of coating and sublayer	Temperature, °C	Test duration, min	Average coefficient of friction	Wear 10 <sup>4</sup> , g/m
80% Ni[Cg] + 20% NiCr (NiCr sublayer)	408–552	140	0.13	1.2
90% Ni[Cg] + 10% Cu (NiAl sublayer)	400–552	160	0.14	1.1
90% Ni[Cg] + 10% Cr (NiCr sublayer)	648–768	70	0.21	2.6

Another problem that can be solved using the composite plasma coatings based on Ni[Cg] is the reduction of mechanical losses in power plants with free-piston engines. Since free-piston engines do not have crankshafts or crank gears, the reverse motion of the piston is provided due to the spring resistance, compressed air, or gravity, the most significant part of the mechanical loss is due to the friction of piston rings in the cylinder, i.e., the free piston Stirling engine is responsible for about 40% of the total losses [3].

At the same time, plasma protective coatings on the parts of the cylinder-piston group and the elements of combustion chamber [4] enable a three to four times reduction in the wear, as well as increase their lifetime, reduce the fuel consumption and the amount of harmful impurities, and increase the engine capacity. It is known [5] that, in piston engines, several approaches to reducing the mechanical losses have been considered: engineering, technological and exploitation. In particular, a comparison of these approaches indicates the effectiveness of the technological approach associated with the development of compositions and methods of the deposition of anti-friction coatings, surface modification [6], and the formation of a regular microrelief on the friction surfaces [7] when using modified motor oils. However, the application of solid lubricating coatings is the most promising because they make it possible to combine the most positive properties of three approaches under consideration, particularly under the condition of the continuous toughening of the operating conditions of the parts and units of engines and power plants [8].

The implemented modeling and analysis of the operating conditions of the parts of cylinder-piston group of a free-piston engine [9] made it possible to formulate the following conditions for laboratory tests for modeling the friction contact: the environment is air; the mixture is oil (fuel) with air, the surface geometry is cylinder-cylinder, the load-pressure in the contact is within  $\leq 1-6$  MPa, and the temperature is up to 250°C when using the oil system and up to 600°C under dry friction using solid lubricant coatings.

An effective direction in the development of the plasma spraying of antifriction wear-resistant coatings with graphite in the case of dry friction is the use of mechanical mixtures of powders. The analysis of the composition of these mixtures for friction pairs of Pratt and Whitney, General Electric, Metso-Sulzer, etc. [10] reveals that the solid, wear-resistant, yet brittle components are most often mixed with more plastic metals and jointly form the coating matrix. In another type of mechanical mixtures, exothermic reacting composite powders (of nickel-aluminum type) are present as a component. Their introduction improves the adhesion strength of the coating with the substrate and of particles with each other in the coating volume. Finally, when the friction unit operates in different variables on time, but repetitive operating conditions (temperatures, pressures in contacts, sliding speeds, etc.), to construct the powder, compositions are the most difficult because forming from them coatings should be equally effective under different operating conditions, which is the subject of special studies.

The mechanical mixtures of powders can be used effectively during plasma spraying when feeding the various powders from separate feeders can be arranged into different points of the plasmatron plasma jet (refractory powders are fed into the plasmatron nozzle or under the nozzle exit section, and fusible powders are fed below along the plasma jet). To obtain a composite mixed coating, the plasma spraying of high-temperature solid lubricant coating simultaneously from two plasmatrons was tested. In this case, the composition was formed in a single spot on the part surface. However, the preliminary mechanical mixing of the powders similar by thermal physical properties and their spraying from a single plasmatron is the cheapest method for obtaining protective materials for the desired operating conditions.

The compositions of the investigated coatings based on Ni[Cg] and their tribotechnical characteristics have been presented in Table 3. The total coating thickness was 190–315  $\mu\text{m}$  at a sublayer thickness of 75–130  $\mu\text{m}$ . Table 3 presents the characteristics of coatings that demonstrated better results with end friction on a UMT-1 friction machine. The samples were rings (the material of the sample with coating and counterbodies is INCO 718 heat-resistant alloy) with an average diameter of 46 mm and a friction width of

2 mm, the pressing pressure is  $\sim 1$  MPa, and speed  $V$  was  $\sim 0.15$ – $1.0$  m/s. The coaxial spiral heater was installed around the rings, making it possible to heat the samples to the desired temperature.

The conducted study revealed the ability to work the plasma coating based on Ni[Cg] under the normal temperature and under the conditions of high temperatures ( $650^\circ\text{C}$  and higher) and at moderate specific pressure, which are typical of the piston ring–cylinder friction pair in the free-piston engine. These studies have made it possible to state that there are considerable scope of reserves to improve the properties of a nickel–graphite coating due to the use of alloying additions, to optimize the composition of coatings based on Ni[Cg], and to improve the technology of their production.

Thus, the results obtained in the work for the study of wear resistance of composite coatings formed by plasma spraying of graphite powder cladding with nickel made it possible to study, implement, and recommend their application under the desired operating conditions, which consist of friction units of aircraft engines and power plants of vehicles based on free piston engines.

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