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MODERN METHODS OF HIGH-ENERGY MODIFICATION OF THE STRUCTURES OF MULTILAYER PLASMA COATINGS

The article considers in detail modern methods of high-energy modification of the structure with subsequent improvement of the properties of the obtained coatings from self-fluxing powders based on iron. Plasma wear-resistant coatings made of materials based on austenitic steels, fabricated by diffusion-alloying with subsequent modification using laser radiation, have been studied. The microstructure of the processed plasma coatings is characterized by a number of features, during high-energy processing, the formed plasma coatings are remelted (to a large extent, remelting correlates with the values of the laser exposure parameters), the formed coatings after processing have a more homogeneous structure and a finely dispersed structure. Conducted studies on the distribution of the microhardness of the applied coating over the depth of the melted layer. To analyze the behavior of laser-treated coatings during operation, we studied the processes of deformation and internal stresses in them. The issues of hardening of wear-resistant plasma coatings during the processing of materials based on powders based on diffusion-alloyed austenitic steels with the addition of molybdenum and molybdenum disulfide are considered and the technological parameters of hardening high-energy treatment of sprayed coatings are optimized.

Keywords: *plasma coatings, compression plasma flows, treatment distance, formed structures, surface layers, formed wear-resistant coatings.*

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СОВРЕМЕННЫЕ СПОСОБЫ ВЫСОКОЭНЕРГЕТИЧЕСКОГО МОДИФИЦИРОВАНИЯ СТРУКТУР МНОГОСЛОЙНЫХ ПЛАЗМЕННЫХ ПОКРЫТИЙ

В статье подробно рассмотрены современные способы высокоэнергетического модифицирования структуры с последующим улучшением свойств полученных покрытий из самофлюсующихся порошков на основе железа. Исследованы плазменные износостойкие покрытия из материалов на базе сталей аустенитного класса, изготовленные способом диффузионно-легированния с последующей модификацией при помощи лазерного излучения. Микроструктура обработанных плазменных покрытий характеризуется рядом особенностей, при высокоэнергетической обработке происходит переплавление сформировавшихся плазменных покрытий (в значительной степени переплавление корелируется с величинами параметров лазерного воздействия), сформированные покрытия имеют после обработки более гомогенную структуру и мелкодисперсное строение. Проведенные исследования по распределению микротвердости нанесенного покрытия по глубине оплавленного слоя. Для анализа поведения обработанных лазером покрытий при эксплуатации, исследовали процессы деформации и внутренние напряжения в них. Рассмотрены вопросы упрочнения износостойких плазменных покрытий при обработке компрессионной плазмой материалов на основе порошков на базе диффузионно-легированных аустенитных сталей с добавлением молибдена и дисульфида молибдена и оптимизированы технологические параметры упрочняющей высокоэнергетической обработки напыленных покрытий.

Ключевые слова: *плазменные покрытия, компрессионные плазменные потоки, дистанция обработки, сформованные структуры, поверхностные слои, формованные износостойкие покрытия.*

1. Introduction

One of the ways to effectively modify the structure of plasma coatings without changing the properties of the base is their treatment with highly concentrated energy flows. The main types of such processing of coatings include: melting of the formed coatings using a plasma installation; application of compression plasma pulses; reflow using a laser beam. An increase in the efficiency of self-fluxing coatings applied by the plasma method based on powder materials based on diffusion-alloyed austenitic steels with the addition of molybdenum and molybdenum disulfide can be carried out by controlling the properties of their individual sections. This contributes to the optimal combination of the properties of various sections of the obtained coatings, which should correspond to the functional purposes of the materials deposited by the plasma. Nevertheless, the technologies for their creation have prospects for further improving the properties of the formed materials as a result of the use of methods for processing them using highly concentrated energy flows. Using layer-by-layer processing of wear-resistant coatings deposited using a plasma installation with short-term pulses of compression plasma flows using different levels of input energy, it is possible to create the possibility of forming certain structures with a controlled and decreasing total porosity from the upper outer layers of the obtained coatings to the base (substrate). This type of treatment contributes to an increase in their oil-retaining capacity and wear resistance of the formed friction surfaces to an additional combination with an increased cohesive and adhesive strength near the boundary layers. On the basis of statistics on the values of limiting wear variations for operated parts, for which the technologies created by us are intended, the thickness of the layers applied by the plasma installation was taken to be about 0.5 - 0.6 mm.

2. Methods for high-energy modification of the structures of multilayer plasma coatings

Various variants of plasma melting of the obtained coatings were carried out at the Institute of Physics and Technology, Siberian Branch, Russian Academy of Sciences, Tomsk. The self-fluxing nickel-based powder alloy (Ni-Cr-B-Si–Fe-Al-C) was chosen as the coating material under study. The deposition of the coatings under study and their melting with the help of nitrogen plasma were carried out on a universal installation UPU-3D in the optimal processing mode in air. [1]. After the plasma reflow process, the size of the main phase of the resulting solid solution increases in the coatings with a decrease in microhardness. At the same time, their porosity decreases and macrohardness increases. All these factors (decrease in porosity with activation of diffusion processes) during plasma treatment contribute to obtaining a very dense coating with increased characteristics of cohesive and adhesive strength. The main disadvantage of this technology is the presence of a zone of thermal influence on the metal of the main product, therefore, as a rule, overall and thick-walled coatings are subjected to melting.

The disadvantage described above is not typical when processing the formed coatings with impulses of a compression plasma jet. As a rule, a plasma injector equipped with a coaxial system of electrodes serves as a source of pulsed impact flows. Such treatment was carried out on the surface of a wear-resistant coating ((Ni-Cr-B-Si-Fe) of rotor blades (IMM NAS of Ukraine, Kiev). Nitrogen served as a plasma-forming gas, while the stored energy in the capacitive storage used was was of the order of 9 × 103 J. During high-energy treatment of coatings during a short-term exposure to a plasma jet of about 800 μs, the resulting heat flux cannot propagate to a very large depth. Therefore, as a result, from the surface, the initial structure of the resulting coating has the form of a melt with a thickness of about 10 μm. As a result, the formed transformed layer is evenly distributed and at the same time closes the outlets of the pores. and the occurrence of surface fragmentation. Therefore, plasma coatings become more efficient and operable in high-temperature operation. The impact of a series of pulses during processing with compressive plasma flows allows layer-by-layer processing of sprayed coatings containing pores with their gradual melting over the entire thickness due to an increase in thermal conductivity of the previous layers and predominant overheating of the untreated subsequent deep layers [1–3]. This makes it possible to significantly increase the performance of the most used wear-resistant plasma coatings. This possibility is realized due to the formation of the structure of surface layers with increased wear resistance and the production of layers boundary with the substrate digging with high characteristics of cohesion and adhesion.

The method of pulse-plasma treatment of applied hard coatings has been patented by OAO "Cherepovets steel-rolling plant" [4]. It is a hardening of the working surface by saturation with carbide-forming elements using plasma pulses. Analyzing the process of pulsed plasma treatment, it must be stated that it belongs to the little-studied processes for processing coatings from composite wear-resistant powder materials.

In the field of plasma deposition of functional coatings, including self-fluxing coatings, processes that use laser radiation treatment are increasingly used [5]. These include laser (such as surfacing and surface modification), as well as hybrid ones (laser-plasma formation of coatings, laser processing combined with high-frequency heating). Usually, when implementing, laser radiation of the technological spectrum is used: gas (CO2 lasers), solid-state (Nd: YAG lasers), diode and fiber [6]. Taking into account the time chronology of the appearance of the technological capabilities of the laser radiation process, the technologies can be decomposed in the following sequence: laser heat treatment, alloying with the help of a laser, deposition and surfacing, and also hybrid welding.

The process of laser heat treatment is associated with the action of a focused laser beam on the workpiece and can be carried out both with and without melting of the hardened surfaces. The latter case requires a pre-applied coating that absorbs laser radiation [7–11]. With laser melting, by changing the parameters of the radiation modes and optimizing them, the minimum diffusion of elements from the base (substrate) is obtained, a dense coating is formed with a melted smooth surface, a rather dispersed microstructure and adhesive strength required for the high-quality operation of the treated parts. Laser melting takes place in Russia and practically in all developed countries of Western Europe, the USA, China, and Japan. Examples of the use of lasers include patents (a method for obtaining a protective coating on a product from a heat-resistant (heat-resistant) alloy [12] and a method for processing friction surfaces [12]), as well as a German patent (a process for manufacturing segment-shaped cutting coatings [13]).

The structure of the layers obtained after melting by laser radiation is characterized by the absence of oxide inclusions and pores, as well as dispersion. The resulting melted zone is characterized by a lower microhardness compared to the plasma spraying process. The character of the depth distribution of the obtained melted zone of alloying elements is uniform, except for the boundaries of the melting zone. This is an essential feature inherent in laser processing. It is provided by the short-term melting of the sprayed layer and subsequent cooling at high rates, which initially contributes to the preservation of the entire spectrum of alloying elements that were contained in the pre-sprayed coatings, and their fairly uniform distribution in the volume after the surfacing process. The known shortcomings of the laser processing process include a low coefficient of conversion of the beam energy into thermal energy and a low productivity of the process.

The main advantages of this process include local thermal effect (since the size of the heating spot is usually in the range of 0.1 ... modification to a depth of several micrometers to a level of 1.0…3.0 mm [14]. The resulting grain refinement contributes to the hardening of metals without the formation of solid phases in them. Certain disadvantages are also characteristic, such as the formation of internal stresses in the hardening tracks, which can lead to the formation of cracks, the likelihood of pore formation during the hardening process with reflow, which is associated with the release of gases due to the burnout of non-metallic inclusions located in base metal [15, 16].

The process of laser alloying involves the creation on the treated surface of a rather small (on the order of d = 0.2 ... 0.6 mm and a depth of 0.1 ... additives [17, 18, 19]. As a result, when the heat source moves relative to the treated surface, a layer with new physical and chemical properties is formed on it. The main advantages of the laser alloying process include [20, 21, 22, 23] the ability to alloy surfaces to a depth of about 2–3 mm during the formation of both chemical compounds and the possibility of creating solid solutions from alloying elements in the structure of the metal itself; obtaining the necessary structures with a fairly high dispersion with a minimum heat-affected zone (HAZ), due to the minimization of thermal effects on the substrate; low residuale deformations. The main disadvantages of this process include the formation of pores, splashes and fistulas, due to the supply of alloying materials (primarily gaseous) to the melt bath.

References [24] considered the effect of laser remelting on the microstructure and corrosion resistance of a plasma-sprayed Fe-based coating deposited by plasma spraying. Then the coatings were further processed by laser melting to improve their microstructure and properties. Corrosion resistance in solutions of 3.5 wt.% NaCl and 1 mol/l HCl of sprayed and laser-fused coatings was evaluated using electrochemical polarization analysis. It was found that the laser remelted coating contains much more amorphous (nanocrystalline) grains than sputtered coatings, which is due to the lower cooling rate in the laser remelting process compared to the plasma spraying process. The results of electrochemical polarization showed that the laser-remelted coating has a higher corrosion resistance than the sputtered coating due to its dense structure.

It is well known that amorphous metallic materials can be obtained by rapid quenching. Therefore, for the manufacture of amorphous metal coatings, several coating technologies with a high cooling rate during processing are used, such as laser surface treatment [25], high-speed sputtering [20], and air-plasma sputtering [10]. Air-plasma spraying is considered as a simple, versatile and effective coating method in both scientific and industrial fields due to its ability to produce much denser, stronger and better coatings [11].

However, pores in a sprayed coating are a typical feature. Corrosion of Fe-based amorphous coatings tended to form around pores [26]. The porosity of the coating made by air-plasma spraying is about 3-5%. Laser processing methods are successfully used to reduce the porosity of sprayed coatings. It was reported that amorphous and nanocrystalline composite coatings can be obtained by laser cladding and remelting [27]. The cooling rate of the laser remelting process was usually about 103 K/s, and the critical cooling rate for the formation of the Ni-Fe-B-Si-Nb amorphous phase was about 233 K/s.

The source [28] prepared Fe-Co-B-Si-Nb coatings on the surface of low-carbon steel using high-power laser melting using [(Fe0.5Co0.5)0.75B0.2Si0.05]95.7Nb4 powder. The extremely high cooling rate of laser remelting is ideal for maintaining or developing a non-equilibrium microstructure, including producing a solid solution with an amorphous state on a chosen crystalline substrate. The source [29] reports the presence of an amorphous phase on the surface subjected to laser remelting of cooled (ledeburite) cast iron with an average hardness of 1200 HV. Therefore, it is believed that laser remelting is a promising technology for increasing the proportion of the amorphous phase and eliminating coating porosity defects.

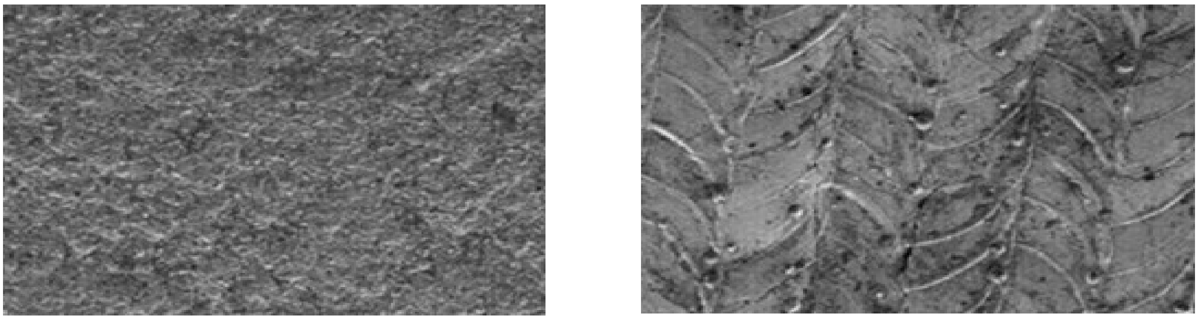
Fe-based metal alloy powder (Beijing SunSpraying Technology Co., Ltd., China) was used as raw material. The Fe-based powder was prepared by gas spraying under an argon atmosphere after the base alloy was melted in a medium frequency vacuum induction furnace and passed through a 160 mesh sieve. The composition of the Fe-based powder is shown in table 1.

Table 1. Chemical composition of iron samoflux

Element	Mo	Cr	Ni	P	Si	B	C	Fe
%, weight	3,4	7,3	2,63	6,34	2,41	0,82	3,84	основа

Iron-based coatings were deposited on the surface of St45 after sandblasting by plasma spraying in the atmosphere. A GP-80 air plasma spraying system was used to obtain coatings with a thickness of about 300 μm. Argon and hydrogen were used as plasma-forming gases. The pressure of both argon and hydrogen was 0.7 MPa during sputtering. The flow rate of argon is 60 l/min, and that of hydrogen is 6 l/min. Argon was used as the powder feed gas, and the powder feed rate was 10 g/min. The power of the plasma jet for coating is at the level of 25 kW (500 A / 50 V). After sputtering, the deposited coatings were remelted with a laser system (HWLW-300A). A high scanning speed of 8000 mm/min was used to remelt the coating at a power of 2 kW. Such parameters give a large width and depth of the remelted layer and lead to a high cooling rate in the remelted layer. During laser processing, a continuous flow of argon gas was maintained to prevent oxidation of the molten pool. The microstructure of the coatings after plasma spraying and laser remelting was characterized by scanning electron microscopy (SEM, S-4800). The phase composition of the powder and coating was analyzed using X-ray diffraction (XRD, D/max 2500PC, Rigaku, Japan) with Cu Kα radiation (λ=1.5418 Å) in the range of 20-80° (2θ) [30].

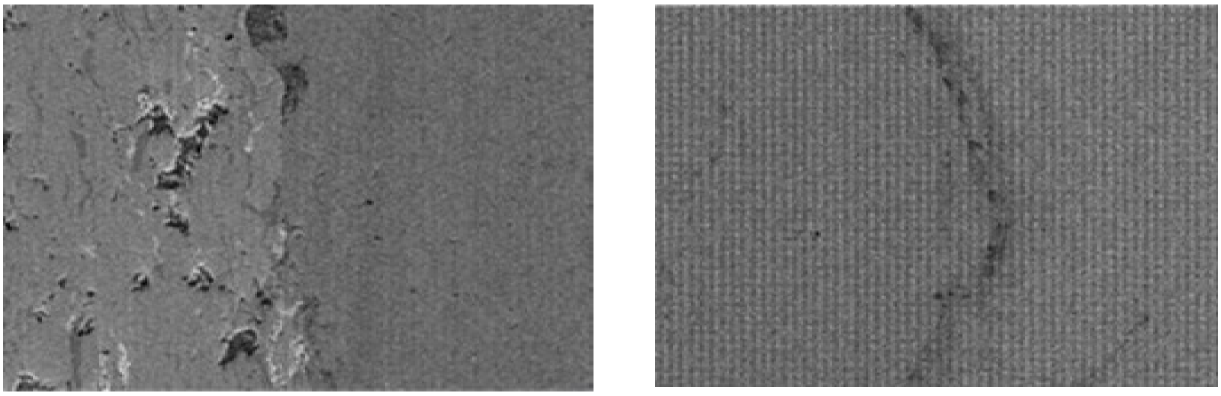
Figure 1 shows the surface morphology of the deposited and laser-treated coatings. As can be seen from Figure 1a, there are many pores on the surface of the deposited coating formed by plasma spraying. This is a typical structure of a plasma-sprayed coating due to the overlap of molten particles. Figure 1b shows the topography of the laser treated coating. It clearly shows the strip of laser scanning of the tracks and the effect of an intense combination of tracks, the scanning traces have specific relief features - there are depressions between two overlapping tracks. Moreover, some small pores also appear in the remelted coating, as shown in Figure 1b. In the process of laser reflow, gases enter the melting bath and, as the temperature decreases, they are released and form pores.



a b

Figure 1. Surface SEM images of coatings obtained by plasma spraying (a) and laser melting (b)

Figure 2a shows the structure of the cross section of iron-based coatings obtained by air-plasma spraying. Generally, all specimens exhibit the typical lamellar structure in plasma sprayed coatings, where sprayed powders deform and solidify when they hit the substrate surface to form splashes. In iron-based coatings, there are many pores and microcracks in the plane. Figure 2b shows the SEM image of the cross section of the coating melted by the laser near the upper surface zone. After laser remelting, a melting depth of 250 μm was observed in the coatings. Compared to Figure 2a, it can be seen that laser melting dramatically changes the microstructure of the coating. There is no lamellar structure in the laser melt and there are fewer pores.



a b

Figure 2. SEM images of the cross section (a) of sputtered and (b) laser-fused coatings

Figure 3 shows X-ray diffraction patterns of a spray-on and laser-melted coating. With the plasma spray coating, the X-ray diffraction pattern shows a broad halo peak at a diffraction angle of 44.8° (2θ), which is a typical characteristic of an amorphous structure. However, the laser remelt coating pattern shows some sharp peaks, which means that crystals are also formed in the remelted layer. Crystalline peaks are identified as the Fe2B phase and the body-centered cubic phase of α-Fe. Laser remelting was used in the surface treatment of amorphous and iron-based coatings obtained by air-plasma spraying. The microstructure of coatings changes during laser treatment with a melt. Laser remelting affects not only the amount of amorphous phase, but also the shape and distribution of crystallization grains. Although the amount of amorphous phase is reduced during the laser remelting process, the dense structure resulting from laser remelting plays an important role in improving the characteristics of the resulting coating.

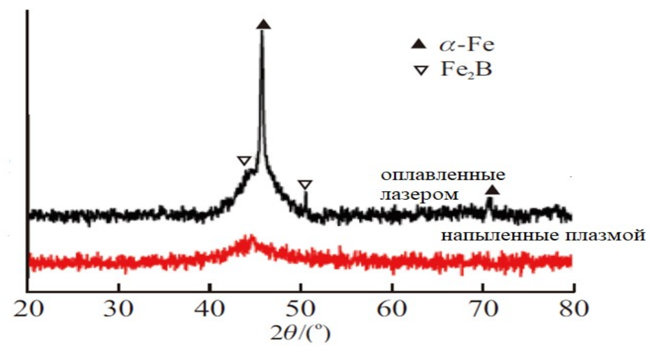
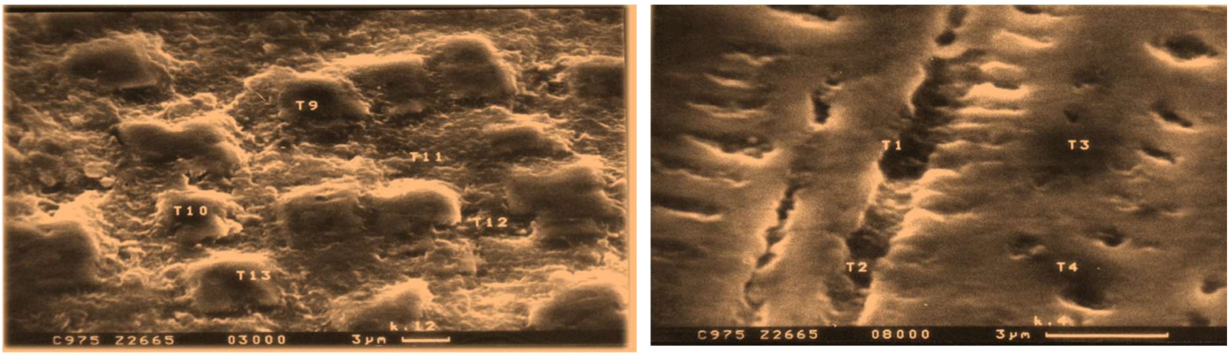


Figure 3. X-ray patterns of sprayed and laser-melted coatings

The source [31] is devoted to the study of the influence of the high-energy laser treatment of plasma coatings from self-fluxing powders based on iron after the process of reflow with modifying coatings on their properties. The samples for research were made of steel grade 40Kh. Iron-based self-flux PR-Kh4G2R4S2F was used for the spraying process. To apply a sublayer of the coating used installation for plasma spraying in air UPU-3D modes: the thickness of the formed layer 0.2 mm, I=250 A; distance - 120 mm; V=80V; nitrogen consumption = 35 l/min, self-fluxing coating and alloying elements in the form of B4C powder coatings were applied using adhesive lubricant with a thickness of 0.09–0.11 mm [31, 32, 33, 34]. Then, the process of melting the resulting composition was carried out using LGN-702 (continuous laser) with the following technological parameters: overlap coefficients k1=0.8 and k2=1.2; laser exposure power N=800 W; beam movement speed v1-5=0.83; 1.67; 2.50; 3.33; 5.00×10-3 m/s; laser beam diameters 1.0×10-3 m and 3.0×10-3 m.

The samples were processed in two modes - soft (coated sample No. 2) and hard (coated sample No. 1). The measurements were carried out in the interdendritic space and along the body of the dendrite (Figure 4 a, b). In the mild mode, the formed structure is characterized by a supersaturated solid solution with inclusions of borides and carbides in it. In the hard mode, the structure consists of small dendrites, with a predominant orientation in the direction of the heat sink obtained.



a b

Figure 4. Structures of the formed coatings from samoflux based on iron ПР-Х4Г2Р4С2Ф alloyed with boron carbide after reflow by a continuous laser: a - laser movement speed - 5·10–3 m/s; b - 0.83·10–3 m/s.

The decisive role in changing the technological parameters of the formed coatings (chemical composition, microhardness and microstructure) was played by laser processing modes. The obtained structures turned from dendritic into supersaturated ones with boride and carbide precipitates with an increase in the speed of the laser beam. In the developed coating, with a decrease in the silicon content, the chromium content increased, and the microhardness increased.

At an increased speed when scanning the laser beam (Figure 4 a), excess carbide and boride precipitates with phase sizes of the order of 5-8 μm are visible in the resulting structure. In the transition zone between the resulting coating and the base, the microhardness was 5.84-6.13 GPa, while the microhardness of the base became at the level of 4.12 GPa. The presence of silicon decreased to values of 0.87–1.23%, which led to a decrease in the microhardness parameter to values of 6.77–7.95 GPa, and the amount of chromium in the solid solution decreased to 3.04–4.12%. A 2.5-2.7-fold decrease in silicon and a sharp (1.5-fold) increase in the content of the chromium element in the carbide-boride phase supersaturated during processing (Figure 4 b) were noted, while the microhardness of this phase increased to values of 11.04- 15.45 GPa.

3. Conclusions

The main trends in the development of hardening treatment of formed plasma coatings using highly concentrated energy flows are technologies for exposing them to plasma flow, compression plasma jet pulses, and laser radiation in order to obtain melted and densified materials after deposition, with modification of their structure. The treatment of very local volumes of the formed coating with high-energy radiation at high rates of heating and cooling processes makes it possible to modify the structure of the deposited material with its strengthening, preferably without the formation of a significant heat-affected zone on the base metal of the product, without undesirable changes properties of the base (substrate). In the field of plasma deposition of functional coatings, including self-fluxing coatings, processes are increasingly used that use high-energy sources, usually laser melting. The highly concentrated modification of the coatings leads to a significant hardening and compaction of the outer layer, while reducing the roughness and, consequently, the uneven heating of the coating, and also eliminates the general porosity in the near-surface layer.

With laser melting, by changing the parameters of radiation modes and optimizing them, it is possible to create the necessary structure in the coating being formed with the creation of a dense coating with a melted and smooth surface, with high-quality adhesive strength for the operation of machined parts, with obtaining a fairly dispersed microstructure and minimal diffusion of elements from the substrate. High-energy sources of energy during the subsequent processing of the formed gas-thermal coatings have their own advantages and features: the ability to control parameters that ensure the regulation of the structure of near-surface layers and their technological characteristics (geometric dimensions of the treated areas, their roughness, hardness, wear resistance, etc.), the use of locality and concentration of energy supplied to the coating being processed and make it possible to process only the near-surface layer of the coating without unnecessary heating of the entire volume with a violation of its properties and structure. The disadvantages include the redistribution and change of stresses in the treated coating. Optimization of processing modes comes down to determining the required radiation powers and coating thicknesses.

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