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СТРУКТУРНО-ФАЗОВЫЕ СОСТОЯНИЯ ПЛАЗМЕННОЙ БЫСТРОРЕЖУЩЕЙ НАПЛАВКИ ПОСЛЕ ОТПУСКА И ЭЛЕКТРОННО-ПУЧКОВОЙ ОБРАБОТКИ

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Аннотация. Методами сканирующей и просвечивающей электронной микроскопии проанализированы структурно-фазовые состояния и дефектная субструктура плазменной наплавки нетоковедущей порошковой проволокой в среде азота быстрорежущей стали Р2М9 на подложку из среднеуглеродистой стали 30ХГСА в исходном состоянии, после высокотемпературного отпуска и электронно-пучковой обработки. Электронно-пучковую обработку проводили при следующих параметрах: энергия ускоренных электронов 18 кэВ, плотность энергии пучка электронов 30 Дж/см^2, длительность импульса 50 мкс, частота следования 50 Гц, количество импульсов 5. Сформированный наплавленный слой толщиной ~ 5 мм имеет карбидную структуру каркасного типа, которая не разрушается при последующих отпуске и облучении импульсными электронными пучками. В исходном состоянии после наплавки и после отпуска присутствуют карбиды состава МС, M_6C, $M_{23}C_6$ и M_7C_3. После электронно-пучковой обработки основным карбидом, формирующим каркас, является карбид состава M_6C. Плазменная наплавка сопровождается формированием мартенситной структуры, по границам и в объеме кристаллов мартенсита выявлены наноразмерные включения второй фазы, имеющие следующий состав: MoC, Mo_2C, M_6C. Их относительное содержание составляет 34 масс. %. в исходном состоянии, 33 масс. % после отпуска и 19 масс. % после электронно-пучковой обработки.

Ключевые слова: наплавленный слой, электронная микроскопия, переходная зона, закалка, импульсный электронный пучок, структура, свойства.

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Original article

STRUCTURE-PHASE STATES OF HSS DEPOSITED LAYER AFTER TEMPERING AND ELECTRON BEAM TREATMENT

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Abstract. The structural-phase states and defect substructure of plasma surfacing with non-current flux-cored wire in the nitrogen medium of R2M9 high-speed steel on the substrate of medium-carbon steel 30HGSA in the initial state, after high-temperature tempering and electron beam treatment were analyzed using scanning and transmission electron microscopy. Electron beam processing was carried out with the following parameters: the energy of accelerated electrons is 18 keV, the energy density of the electron beam is 30 J/cm^2, the pulse duration is 50 microseconds, the repetition frequency is 0.3 Hz, the number of pulses is 5. The formed deposited layer with a thickness of ~5 mm has a frame-type carbide structure, which is not fractured during subsequent tempering and pulsed electron beams irradiation. Carbides in compositions MC, M_6C, $M_{23}C_6$ and M_7C_3 are present in the initial state after surfacing and after tempering. After electron beam treatment, the main carbide forming the frame is M_6C carbide. Plasma surfacing is accompanied by the formation of martensitic structure; nanosized inclusions of the second phase, having the following compositions: MoC, Mo_2C, M_6C, were identified along the boundaries and in the volume of martensite crystals. Their relative content is 34 wt. %. in the initial state, 33 wt. % after tempering and 19 wt. % after electron beam treatment.

Keywords: deposited layer, electron microscopy, transition zone, tempering, pulsed electron beam, structure, properties.

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Introduction

Reliability and durability of the working surfaces of equipment and mechanisms are determined mainly by the quality of their protection against wear and corrosion. The most common causes of premature failure of machine and equipment parts are abrasive and impact-abrasive wear, cavitation, corrosion, and fatigue processes. This problem is solved by controlled changes in the properties of working surfaces through surfacing that provides the required set of properties [1-4]. Development of coatings with improved performance characteristics under extreme conditions is a fundamental task of great economic importance [5-7].

High performance properties of deposited high-speed steels (HSS) are achieved by special alloying and complex heat treatment, which ensures a certain phase composition [8-12]. High hardness and wear resistance of high-speed steels are due to their alloying with carbide-forming elements: tungsten, vanadium, molybdenum and chromium. These elements, under certain temperature and time conditions, form particles of the carbide phase in steel, which are the strengthening phase of the material [13].

The use of a shielding and alloying medium of nitrogen during plasma surface treatment, which has significant advantages over other methods, substantially improves abrasive wear resistance, corrosion and impact resistance, and strength due

to the formation of carbonitrides with increased microhardness [14,15]. Plasma surfacing in the nitrogen medium with a non-current-carrying flux-cored wire leads to the production of deposited high-speed steels alloyed with nitrogen and aluminum, which increases their hardness and wear resistance and reduces the cost of surfacing [1,14,15]. Improvement of tribological and mechanical properties and the quality of the deposited surface layer can be achieved by electron beam treatment [16-20]. It provides good damping properties under mechanical and thermal loads and prevents the premature initiation of brittle microcracks.

The analysis of related literature published in Russia and abroad shows that there are very few studies devoted to the physical nature and formation mechanisms of improved HSSs surfacing properties using transmission electron microscopy [1,20].

The purpose of this work is to analyze the results obtained from studying the elemental and phase composition, the state of the defective substructure of the layer deposited on 30HGSA steel with PP-R2M9 flux-cored wire and subjected to high-temperature tempering and electron beam treatment.

Materials and methods

The deposited material was investigated in three states: firstly, after the formation of the deposited layer (hereinafter referred to as the initial state), secondly, after three temperings of samples in the initial state, and thirdly, after three temperings and additional irradiation with a pulsed electron beam.

The samples for research were produced by plasma surfacing using PP-R2M9Yu non-current-carrying flux-cored wire on 30HGSA steel in the nitrogen medium. Chemical composition of 30HGSA steel, % (by weight): 0.3C; 0.9Cr; 0.8Mn; 0.9Si. Chemical composition of R2M9Yu alloy, % (by weight): 0.86C; 4.8Cr; 2.5W, 9.4Mo; 0.5V; 0.85Al; 0.08N; the rest is iron. Plasma surfacing modes did not differ from those described in [1,2,15]. Some samples of 30HGSA steel with a deposited layer of R2M9 alloy were subjected to high-temperature tempering at a heating temperature of 580 °C, holding time – 1 hour, number of temperings – 3. After tempering, part of the samples was subjected to additional irradiation with a pulsed electron beam. Pulsed electron beam treatment was carried out on the SOLO installation (IHCE SB RAS) [19,20] with the following parameters: energy of accelerated electrons – 18 keV; electron beam energy density – 30 J/cm^2, exposure pulse duration – 50 μs, pulse repetition frequency – 0.3 s^{-1}, number of pulses – 5; irradiation was carried out at a residual gas pressure (argon) in the working chamber of the installation – 0.02 Pa.

Studies of the structure and elemental composition of the deposited layer were performed using scanning (KYKY-EM6900 device equipped with an AZtec Live Lite Xplore 30 EDS energy-dispersive microanalysis system) and transmission diffraction (JEM2100 device) electron microscopy [21-23]. The phase composition and state of the crystal lattice of the phases were studied by X-ray diffraction analysis (DRON-8N X-ray diffraction meter).

Results and discussion

Plasma surfacing in the nitrogen medium with PP-R2M9Yu non-current-carrying flux-cored wire on 30HGSA steel led to the formation of a layer with a thickness of 5 mm. The deposited layer has a frame-type carbide structure formed by grains of two dimensional levels [20]. The grains of the first level have sizes varying within the range of (15-35) μm. The grain sizes of the second level vary within the range of (3.5-11) μm. At the grain boundaries, predominantly of the first size level, inclusions of the second phase are located, forming a discontinuous shell. Additional heat treatment did not result in a fracture of the deposited layer frame structure. It should be noted that pulsed electron beam irradiation of the deposited layer polished surface led to the formation of a large-scale (≈900 μm) honeycomb-type structure. This may indicate self-organization of the surface layer structure subjected to high-speed melting and subsequent high-speed hardening.

A lamellar-type structure typical for lamellar martensite is observed in the volume of grains of the deposited layer.

Heat treatment of the deposited layer does not lead to fracture of the lamellar structure. In the volume and along the boundaries of the plates, particles of the second phase are distinguished, the sizes of which vary from 20 nm to 45 nm. Additional treatment of the deposited layer with a pulsed electron beam is also accompanied by the formation of a plate-type structure with numerous nano-sized precipitates of the second phase in the volume of surface layer grains.

The phase composition of deposited layer at the macro level was studied using X-ray phase analysis.

It was found that the main phase of the deposited layer in the initial state and after repeated heat treatments is a solid solution based on α-Fe (bcc iron-based crystal lattice) (Fig 1). Along with the α-phase, the γ-phase (solid solution based on the fcc iron lattice, retained austenite) is present in a small amount (3-5 wt. %) in the deposited layer (in the initial state and after heat treatment). The change in the crystal lattice parameter of the α- and γ-phases depending on the treatment mode, shows that repeated temperings lead to a decrease in the crystal lattice parameter of both phases. This indicates the decomposition of the solid solution followed by the release of carbide phase particles. Additional irradiation of the deposited layer with a pulsed electron beam is accompanied by a significant increase in the crystal lattice parameter of the α-phase, which obviously proves re-hardening of the deposited layer surface. At the same time, pulsed electron beam irradiation is accompanied by a decrease in the crystal lattice parameter of the γ-phase. The latter indicates the decomposition of the solid solution based on γ-iron with the subsequent release of carbide phase particles in the deposited layer surface.

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As mentioned above, the deposited layer is characterized by the presence of inclusions of the second phase. Following the results presented in Fig. 1, it can be stated that repeated temperings do not lead to an increase in the relative content of carbide phase particles. Additional pulsed electron beam irradiation contributes to a significant (1.8 times) reduction in the relative content of carbide phase particles. X-ray phase analysis showed that the deposited layer in the initial state contains carbides in the following compositionы: MC (15 wt. %), M_6C (10 wt. %), M_7C_3 (7 wt. %), $M_{23}C_6$ (2 wt. %). Repeated temperings led to a slight change in the composition and ratio of carbide phases: MS (18 wt. %), M_6C (12 wt. %), $M_{23}C_6$ (3 wt. %), M_7C_3 (traces). Additional pulsed electron beam irradiation of the deposited layer, subjected to repeated temperings, caused significant transformation of the carbide phase, namely, the main carbide is MS (19 wt. %), M_6C (traces).

Transmission electron diffraction microscopy showed that particles of the carbide phase can be divided into two categories according to their size. Firstly, carbide particles, the transverse dimensions of which are a few micrometers. Such particles form a carbide framework. Secondly, in the volume of grains and crystals of lamellar martensite there are particles tens to hundreds of nanometers in size. Micro-X-ray spectral analysis proves that the particles of the carbide framework are enriched predominantly in molybdenum and tungsten atoms.

The analysis of microelectron diffraction pattern, obtained from micron-sized inclusions, indicates that this formation is a carbide of complex composition of the M_6C type.

It was noted above that the second phase particles of nanoscale range are located in the volume and at the boundaries of martensite plates. Studies carried out using mapping methods indicate that these particles are enriched in molybdenum atoms.

Indeed, the analysis of microelectron diffraction pattern obtained from the region of foil containing such inclusions showed that these particles are molybdenum carbide of composition Mo_2C.

Previously it was noted that pulsed electron beam irradiation of the deposited layer does not lead to the fracture of frame-type structure. Micron-sized inclusions located along grain boundaries are enriched, as mapping results show, in atoms of iron, molybdenum and tungsten.

Dark-field analysis with a subsequent indexing of the microelectron diffraction pattern, obtained from the foil region given in Fig. 2a, showed that this inclusion is s carbide of composition M_6C (Fig. 2b).

It was found that the main phase of the deposited layer in the initial state and after repeated heat treatments is a solid solution based on α-Fe (bcc iron-based crystal lattice) (Fig 1). Along with the

α-phase, the γ-phase (solid solution based on the fcc iron lattice, retained austenite) is present in a small amount (3-5 wt. %) in the deposited layer (in the initial state and after heat treatment). The change in the crystal lattice parameter of the α- and γ-phases depending on the treatment mode, shows that repeated temperings lead to a decrease in the crystal lattice parameter of both phases. This indicates the decomposition of the solid solution followed by the release of carbide phase particles. Additional irradiation of the deposited layer with a pulsed electron beam is accompanied by a significant increase in the crystal lattice parameter of the α-phase, which obviously proves re-hardening of the deposited layer surface. At the same time, pulsed electron beam irradiation is accompanied by a decrease in the crystal lattice parameter of the γ-phase. The latter indicates the decomposition of the solid solution based on γ-iron with the subsequent release of carbide phase particles in the deposited layer surface.

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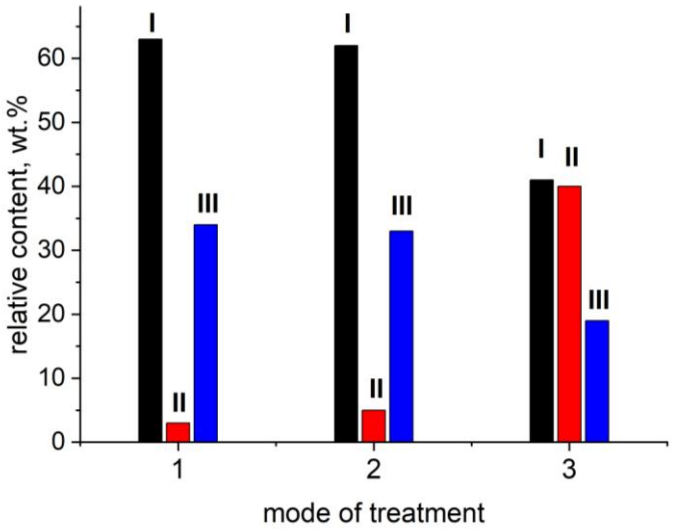

Рис. 1. Относительное содержание фаз, выявленное методами рентгеноструктурного анализа: I – α-Fe, II – γ-Fe, III – карбиды; режимы обработки: 1 - наплавленный слой в исходном состоянии, 2 - наплавленный слой после многократного отпуска, 3 - наплавленный слой после многократного отпуска и дополнительного облучения импульсным электронным пучком

Fig. 1. Relative content of phases revealed by X-ray diffraction analysis: I – α-Fe, II – γ-Fe, III – carbides; processing modes: 1 – deposited layer in the initial state, 2 – deposited layer after repeated temperings, 3 – deposited layer after repeated temperings and additional pulsed electron beam irradiation.

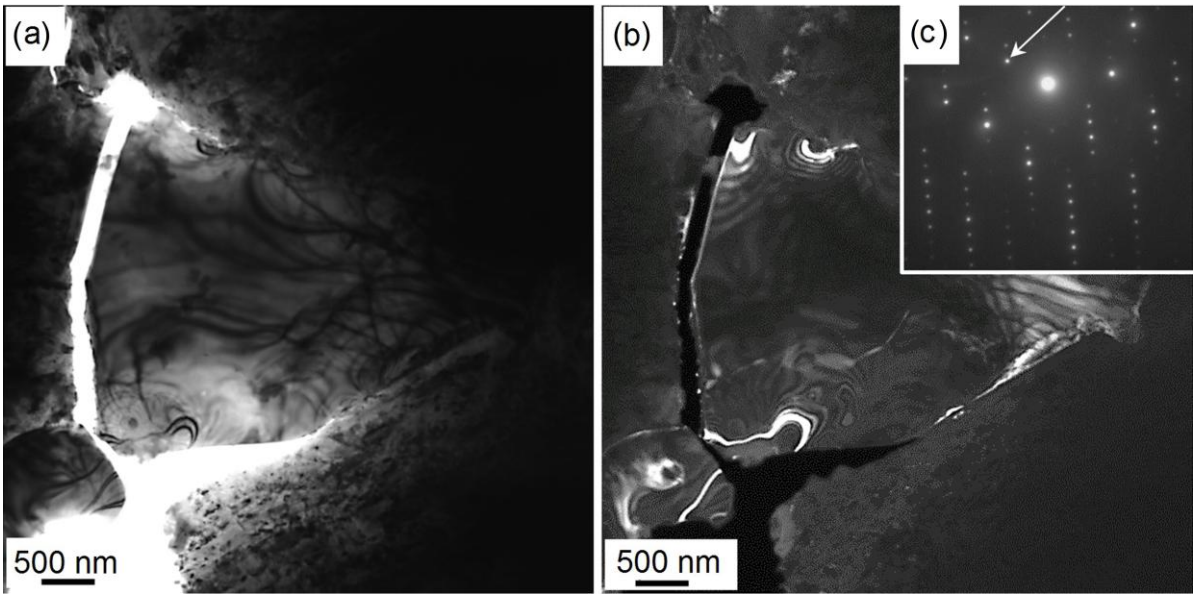

Рис. 2. Электронно-микроскопическое изображение включений микронных размеров, выявленных методами STEM анализа в наплавленном слое после многократного отпуска (а) и дополнительного облучения импульсным электронным пучком (б)

Fig. 2. EM image of the deposited layer structure after repeated temperings and additional pulsed electron beam irradiation: a – bright field; b – dark field obtained in the [511]M_6C reflection; c – microelectron diffraction pattern, the arrow indicates the reflection in which the dark-field image was obtained (b).

Transmission electron diffraction microscopy showed that particles of the carbide phase can be divided into two categories according to their size. Firstly, carbide particles, the transverse dimensions of which are a few micrometers. Such particles form a carbide framework. Secondly, in the volume of grains and crystals of lamellar martensite there are particles tens to hundreds of nanometers in size. Micro-X-ray spectral analysis proves that the particles of the carbide framework are enriched predominantly in molybdenum and tungsten atoms.

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It was noted above that the second phase particles of nanoscale range are located in the volume and at the boundaries of martensite plates. Studies carried out using mapping methods indicate that these particles are enriched in molybdenum atoms.

Indeed, the analysis of microelectron diffraction pattern obtained from the region of foil containing such inclusions showed that these particles are molybdenum carbide of composition Mo_2C.

Previously it was noted that pulsed electron beam irradiation of the deposited layer does not lead to the fracture of frame-type structure. Micron-sized inclusions located along grain boundaries are enriched, as mapping results show, in atoms of iron, molybdenum and tungsten.

Dark-field analysis with a subsequent indexing of the microelectron diffraction pattern, obtained from the foil region given in Fig. 2a, showed that this inclusion is s carbide of composition M_6C (Fig. 2b).

The intragranular structure of the deposited layer irradiated with a pulsed electron beam is characterized by the presence of nanosized particles of the second phase. With the help of the mapping method it was established that these particles are enriched in molybdenum atoms and, in a lesser extent, tungsten atoms.

Using dark-field analysis with subsequent indexing of microelectron diffraction patterns, it was found that these particles are molybdenum carbides of composition MoC and carbides of complex composition M_6C. The sizes of particles having a globular shape are 50-65 nm. Along with these particles, particles with sizes of (3.5-4.5) nm were identified in the volume of plates on dislocations.

It was noted above that pulsed electron beam irradiation of the deposited layer leads to crystallization of the surface layer with the preservation of a significant amount of retained austenite. Transmission electron diffraction microscopy established that the main location of retained austenite is the boundaries of martensite crystals (Fig. 3).

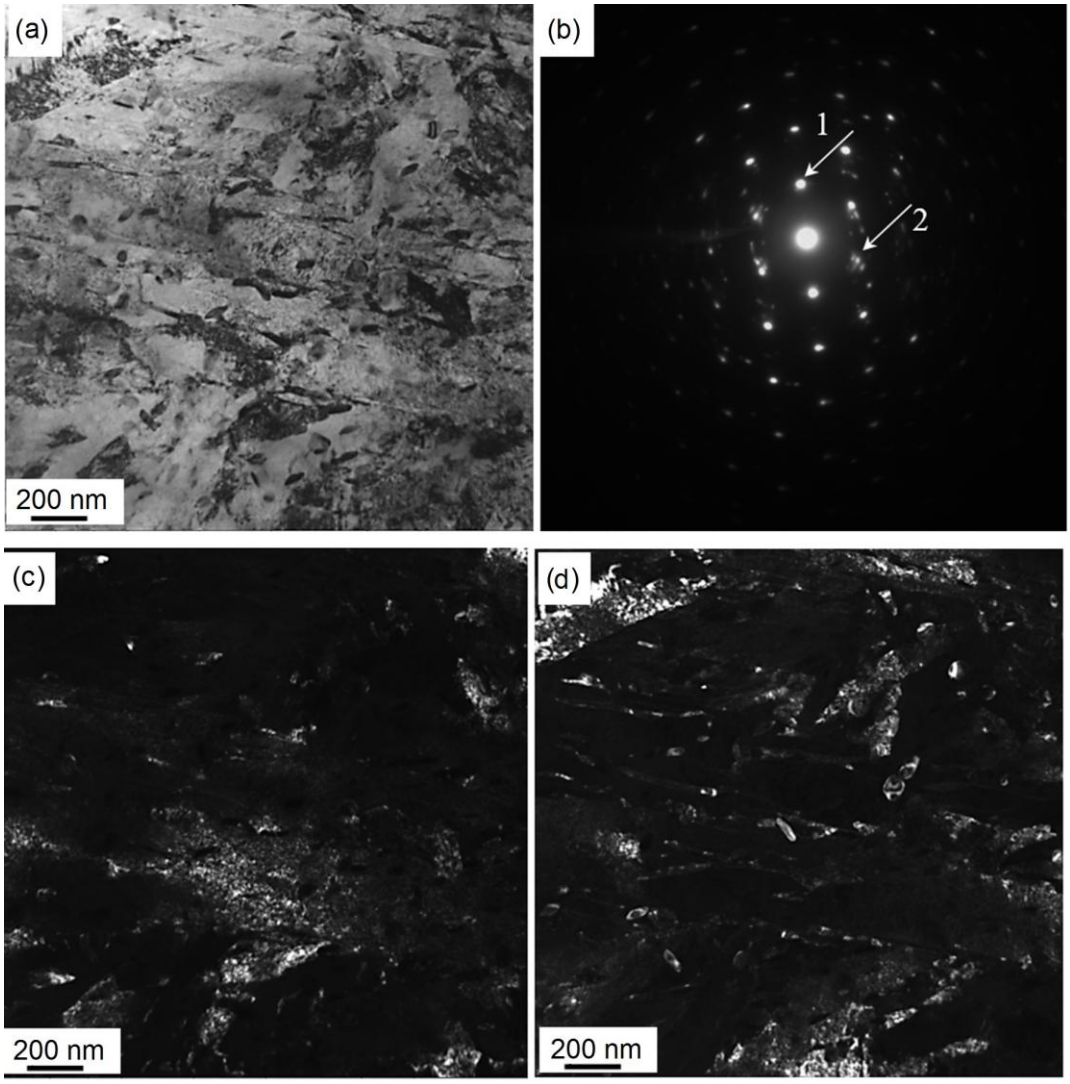

Рис. 3. Электронно-микроскопическое изображение структуры наплавленного слоя после многократного отпуска и последующего облучения импульсным электронным пучком: а – светлое поле; б – микроэлектронограмма; в, г – темные поля, полученные в близко расположенных рефлексах [110]α-Fe + [103]MoC (в) и [110]α-Fe +[002]γ-Fe + [100]MoC (г). На (б) стрелками указаны рефлексы, в которых получены темнопольные изображения: 1 – (в), 2 – (г)

Fig. 3. EM image of the deposited layer structure after repeated temperings and subsequent pulsed electron beam irradiation: a – bright field; b – microelectron diffraction pattern; c, d – dark fields obtained in closely located reflections [110]α-Fe + [103]MoC (c) and [110]α-Fe + [002]γ-Fe + [100]MoC (d). In (b) the arrows indicate the reflections in which dark-field images were obtained: 1 – (c), 2 – (d).

Three high-temperature temperings of steel samples with a deposited layer did not lead to a significant change in the morphology of particles of the carbide phase in the transition zone. As in the initial state, particles of spherical, globular and lamellar shapes are observed in the structure of the transition zone after tempering. The sizes of globular particles vary within a range of up to 100 nm; spherical particles – within a few nanometers.

Conclusion

Plasma surfacing in the nitrogen medium with a non-current-carrying flux-cored wire PP-R2M9Yu on 30HGSA steel formed a layer with a thickness of (4.5-5) mm having a frame-type carbide structure. It was shown that additional heat treatment (repeated temperings, pulsed electron beam irradiation) does not result in a fracture of this structure.

It was established that plasma surfacing is accompanied by the formation of a martensitic structure; nanosized inclusions of the second phase are observed along the boundaries and in the volume of martensite crystals. X-ray phase analysis showed that inclusions of the second phase are carbides, the relative content of which in the deposited layer is 34 wt. %, in the deposited layer after tempering – 33 wt. %, after tempering and additional pulsed electron beam irradiation – 19 wt. %. In the deposited layer in the initial state and the state after tempering there are carbides in compositions MC, M_6C, $M_{23}C_6$ and M_7C_3; after additional pulsed electron beam irradiation, the main carbide of the deposited layer is MC carbide. It was established that, regardless of the heat treatment mode of the deposited layer, the main phase forming the carbide frame is carbide of composition M_6C. Nanosized particles of the carbide phase located in the volume and along the boundaries of martensite plates have the following compositions: MoC, Mo_2C, M_6C.

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