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Research into the possibility of improving the quality of electric arc coatings by nitriding

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Abstract

An effective way to effectively change the characteristics of electric arc coatings made from crushed materials can be achieved by creating high-quality surface balls in them due to the use of different methods. chemical-thermal treatment. Research was carried out on the development and testing of the concept of molding chemical dry coatings based on the combined process of electrode deposition with technological processing of pulsed ion nitriding. It has been shown that duplex (combined) technology, which combines electric arc filing and nitriding, allows one to obtain a significant effect on non-structural properties, increased hardness, heat transfer and wear resistance of the part-coating system with a reduced degree of deformation of the part. An assessment was made of the possibility of moving the electric arc coatings from martensitic steels (40X13, 95X18), ferrite (Sv-08G2S) and austenitic (X18H10T, 12X18H10T) classes. further processing of nitriding.

Keywords: combined methods of applying wear-resistant coatings, electric arc spraying, chemical-thermal treatment, nitriding, wear and corrosion resistance, strengthening protective coatings

Introduction

The use of coatings makes it possible to increase the wear and corrosion resistance of working surfaces of machine parts and mechanisms, in particular ship parts, and so to reduce the costs of alloyed steels and alloys [1].

The coatings application is associated with implementation of a fundamentally new approach, according to which the strength and carrying capacity of a part is provided by its basic material, whereas the resistance to corrosion, wear, and other factors may be increased *via* using hardening protective coatings. There are many alternative methods for producing coatings, from which it is advisable to choose an optimal, easy to implement, and inexpensive one [2]. Of the variety of methods for hardening coating deposition, the most common technologies used to restore and improve the performance properties of parts are gas-thermal spraying techniques [2], among which the cheapest and simplest method is electric arc spraying (EAS), whose current improvement is aimed at modifying and activating the spraying process [3]. A significant increase in the properties of EAS coatings is possible through combining arc spraying with ultrasonic [3], electric-spark [2], laser [10-14], electron-beam [2], and other processing techniques [2]. High wear resistance, hardness, and other surface properties of EAS coatings from iron based alloys can be provided by methods of chemical heat treatment [2]. The use of a combination of techniques for EAS and subsequent chemical heat treatment opens up great opportunities in creating composite coatings with special properties. Methods for improving the quality of electric arc coatings *via* subsequent heat treatment or modification are easily implemented in practice, in particular when part dimensions permit it. Such combined technologies do not require additional expensive equipment and operations, which predetermines a reduction in the cost of hardening processes.

Thus, an effective way to solve the problem of increasing the performance characteristics of EAS coatings from wire materials may be the formation of high strength surface layers through the use of various methods for chemical heat treatment. Combining coating with surface modification and treatment of produced coatings allows the development of new combined methods for surface engineering. Among a large number of techniques for diffusional alloying of surface layers, nitriding methods are notable for high manufacturability, environmental safety, and economic efficiency [20]. Therefore, for the subsequent modification of EAS steel coatings, pulse ion nitriding (PIN) was chosen [15, 16].

The aim of the work was to develop a new combined method of surface engineering for the formation of hardening protective coatings on the basis of combination of EAS followed by treatment with PIN.

Materials and procedures

For coating deposition, an apparatus for activated arc spraying ADN-10 was used. Coating materials were 0.8-2.8 mm diameter wire from steel of the martensitic (40Kh13, 95Kh18), ferritic (Sv-08G2S), and austenitic (Kh18N10T, 12Kh18N10T) grades. A feature of martensitic and austenitic steels of is the ability to phase transformations and structural changes during deposition and treatment of coatings. This allows improvement of physic mechanical and performance properties of hardened surfaces and an increase in their wear and corrosion resistances. The PIN process lasted 2 h in the temperature range 600 - 800 K.

Microstructure was examined on etched and unetched thin sections using a light microscope «MeF-3» (Firm "Reichert", Austria) with magnifications ×100, ×200, and ×500. The microhardness was measured on a Micromet II microhardness meter with a load of 100 g from the coating surface edge to the base through the transition zone. A quantitative stereological analysis of coating porosity was carried out on a certified automatic image analyzer “Mini-Magiscan” (Firm “Joyce Loebl”, England) using the program “Genias 26”. The main stages of the image analysis were: image calibration, image fixation, segmentation, and porosity. The study was performed on a CamScan scanning electron microscope (Oxford Instruments, England) with an X-ray energy dispersive analyzer. The morphology (topography) of the coating surface was examined in the regime of reflected electrons at an accelerating voltage of 10–20 kV.

Research results and discussion

As a result of the research, an assessment was made for possibility to improve the quality of EAS coatings by subsequent PIN. Data on microhardness are summarized in Table 1.

Table 1

The effect of PIN temperature on the microhardness of EAS coatings

Material of coating	Microhardness, GPa, at different temperatures of nitriding, K						
	600	620	650	670	700	720	770
Martensitic steel	8.6	11.3	12.9	14.0	15.9	12.7	11.3
Austenitic steel	5.6	6.2	8.0	8.9	11.1	12.0	11.8

The results of the study of structural parameters and phase composition of ion-modified coatings under various conditions are presented in Tables 2 and 3.

Table 2

Structure parameters of EAS coatings from austenitic steels after PIN

Conditions of PIN		Thickness of layer, μm	Phase composition
AEAS	N^+ 620 K	5-10	α-Fe, γ-Fe, γ_N, Fe_3O_4, ε-$(Fe,Cr)_3N$
	N^+ 670 K	10-15	α-Fe, Fe_3O_4, ε-$(Fe,Cr)_3N$, γ'-Fe_4N
	N^+ 720 K	15-20	α-Fe, Fe_3O_4, ε-$(Fe,Cr)_3N$, γ'-Fe_4N, CrN
	N^+ 770 K	25-30	α-Fe, Fe_3O_4, γ'-Fe_4N, CrN

Table 3

Structure parameters of EAS coatings from austenitic steels after PIN

Conditions of PIN		Thickness of layer, μm	Phase composition
AEAS	N^+ 620 K	3-5	α-Fe, γ-Fe, Fe_3O_4, γ_N,
	N^+ 670 K	3-5	α-Fe, γ-Fe, Fe_3O_4, γ'_N,
	N^+ 720 K	10-15	α-Fe, γ-Fe, Fe_3O_4, γ'_N, γ'-Fe_4N, CrN
	N^+ 770 K	15	α-Fe, γ-Fe, Fe_3O_4, γ'-Fe_4N, CrN

According to the data obtained, as the temperature of PIN increases, the modified layer depth increases as well (up to 50 µm). In addition, the microhardness of the layer significantly increases and, as the result of treatment at 670…720 K, reaches the level of maximum values (14.0…15.9 GPA). The main phases present in the nitrogen-modified layers on martensitic steel coatings are the nitrides ε - $(Fe, Cr)_3N$ and γ′-Fe_4N. After ion treatment at 720 and 770 K, a sharp decrease in the high-nitrogen ε-nitride content and formation of CrN are recorded in nitrides layers, while the layer microhardness decreases to 11,3…12.7 GPA. For the layers formed by PIN on EAS coatings, a relatively high microhardness and a large depth of the dopant penetration are characteristic. In addition, a distinctive feature of nitride layers on the electric arc coating is the presence of particles of the α′′- $(Fe, Cr)_3N$ phase in them along with a reduced content of the γ′-Fe_4N nitride phase and a relatively higher content of CrN.

The increased diffusion permeability of the coatings is caused by high concentration of defects (vacancies, dislocations, pores) contained in them, which were formed under the conditions of very rapid crystallization of molten drops in the course of EAS. At the same time, the presence of the thermodynamically stable chromium-doped Fe_3O_4 oxide in the form of films, separating the sprayed particles, makes diffusion of nitrogen from the upper layer of particles to the underlying layers difficult. The greatest depth of PIN is achieved in EAS coatings with a reduced content of oxide films. PIN of EAS coatings from austenitic steel leads to the formation of modified layers, 3…5 to 15…25 µm thick (Table 3). Therefore, the coatings were formed by activated electric arc spraying (AEAS), providing a porosity of less than 7% (Fig. 1, b).

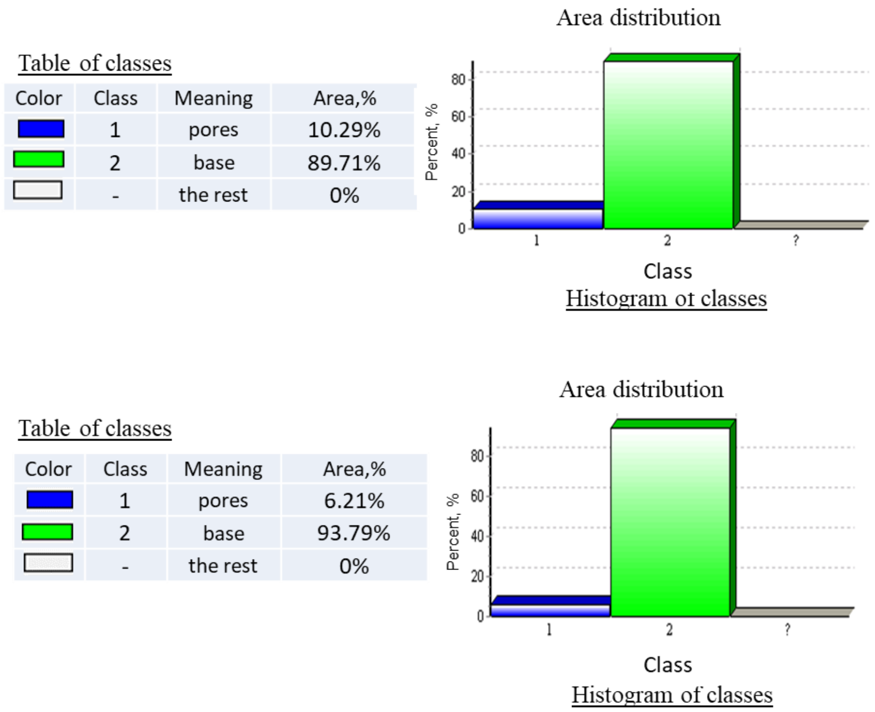


Table of classes

Color	Class	Meaning	Area,%
🟦	1	pores	10.29%
🟩	2	base	89.71%
☐	-	the rest	0%

Table of classes

Color	Class	Meaning	Area,%
🟦	1	pores	6.21%
🟩	2	base	93.79%
☐	-	the rest	0%

Fig. 1. **Porosity distribution in coatings obtained by: (a) EAS and not recommended for treatment by PIN; (b) AEAS and recommended for treatment by PIN**

The studies showed (Table 4) that at a layer density of more than 94%, this effect does not arise.

Table 4

Results for topography of layer surfaces after PIN at 580 K

Method of spraying	Material for spraying	Porosity of layer, %	Surface microtopography
EAS	Ferritic steels	≈ 10 ≥ 8	Bulging Bulging
Activated EAS		≈ 6 ≈ 5	smooth and unchanged smooth and unchanged
EAS	Martensitic steels	≈ 10 ≥ 8	Bulging Bulging
Activated EAS		≈ 6 ≈ 5	smooth and unchanged smooth and unchanged

The main reason for the relatively small depth of nitrogen saturation of electric arc coatings (15…30 µm) compared with that for cast steels is the presence of a large number of oxide films in the coating, which act as a barrier to the PIN process. Oxides are inevitably formed both during the flight of molten particles and in the course of their crystallization on the surface of a part. For the same reason, it is almost impossible to strengthen coatings with porosity over 6% without formation of defective areas, since their pore walls are covered with a thin layer of iron oxides.

The formation of oxide films can be eliminated *via* spraying steel wires with the products of propane and air combustion, that is, *via* using AEAS. The consumption of propane and air combustion products during AEAS is 30–40 m^3/h. In the course of the subsequent heating up to the nitriding temperature, which is in the range of 530–640 K (depending on the steel grade), the process of hardening and release of gaseous products to vacuum occur. It was established experimentally that the higher the nitriding temperature, the shorter the time interval between the temperature of PIN hardening and the start of surface saturation with nitrogen (Fig. 2). The samples after chemical heat treatment were cooled with the container to room temperature.

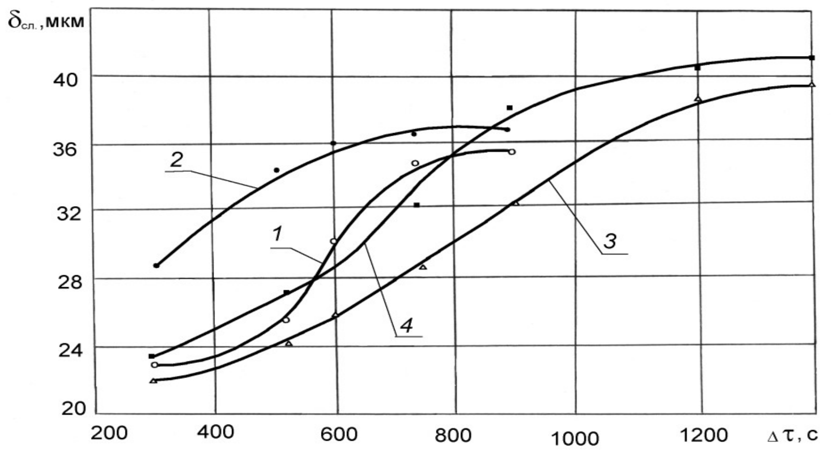


Fig. 2. Dependence of the nitrided layer thickness on the time interval between heating up to PIN temperature and the start of surface saturation with nitrogen for coatings from martensitic steels (1,2) and austenitic steels (3,4) at: (1) 680 K; (2) 710 K; (3) 50 K; (4) 680 K

As a result of tribological tests (pressure 0.64 MPa, dry friction) of coatings after various PIN modes, it was established that an increase in the PIN temperature for 40Kh13 steel coatings leads to a sharp increase in their wear resistance. The coatings treated with nitrogen ions at 670–770 K (Fig. 3) have the highest wear resistance. The wear rate of the counterbody (hardened steel 60G, HV = 78–80 GPa) slightly decreases during the EAS–PIN transition.

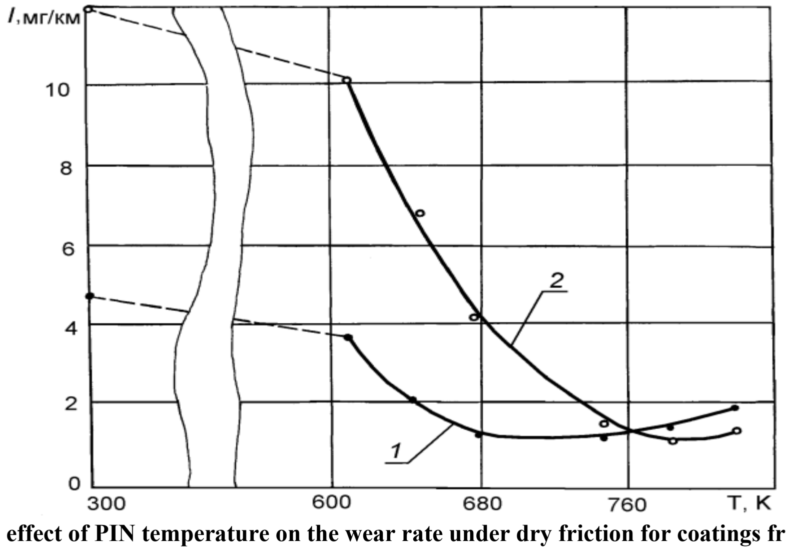
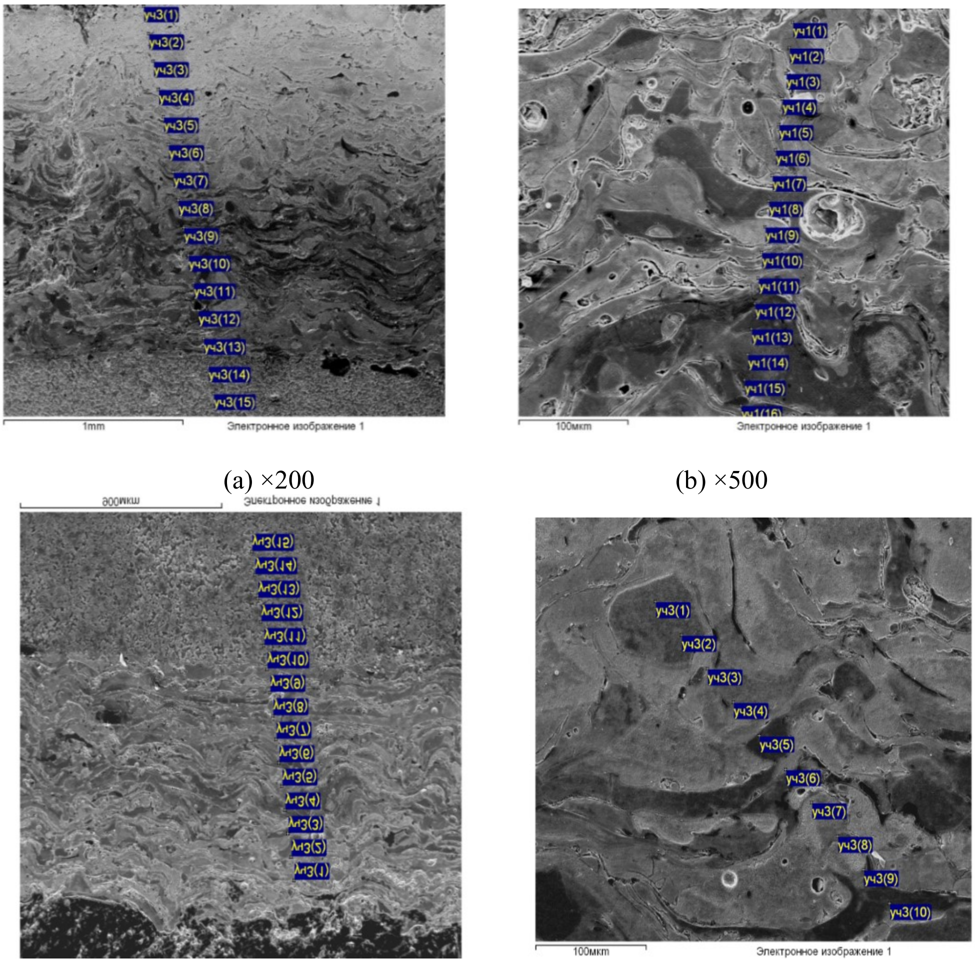


Fig. 3. The effect of PIN temperature on the wear rate under dry friction for coatings from martensitic steel (1); austenitic steel. (2)

Nitriding of Kh18N10T steel coatings at 500–520 K, which results in the formation of a modified 3–5 μm thick gradient layer, was not accompanied by increase in the wear resistance of steel surface under conditions of contact interaction without lubrication. At higher temperatures (670, 720 and 770 K), the wear resistance of coatings increases markedly. The increased wear resistance of EAS coating nitrided at 770 K is due to its significant depth and high microhardness.

It should be noted that a nitrogen-modified layer has an uneven thickness. The areas in the form of layers are detected in the coating depth and at the boundary with the substrate. The revealed morphology of modified interlayers in the depth of the deposited layers indicates a boundary mechanism of diffusion of interstitial atoms in the coatings. The results of metallographic analysis also indicate the preservation of oxide films in a modified coating from steel 40Kh13. By changing the process temperature as well as the potential ratio of nitrogen, one can control the depth of diffusion layer and its hardness. It was established (Fig. 4 c, d) that after nitriding of electric arc coatings from steel 40Kh13, an anomalous structure is formed, which was not before observed in these systems. It is a composite type structure consisting of a steel matrix and solid nitrides, the appearance of which is due to intense diffusion along the boundaries of conglomerates of deformed wire particles. The microhardness of the diffusion layers on coatings is higher than that of the similar layers on cast steel 40Kh13 and reaches 10 GPA.

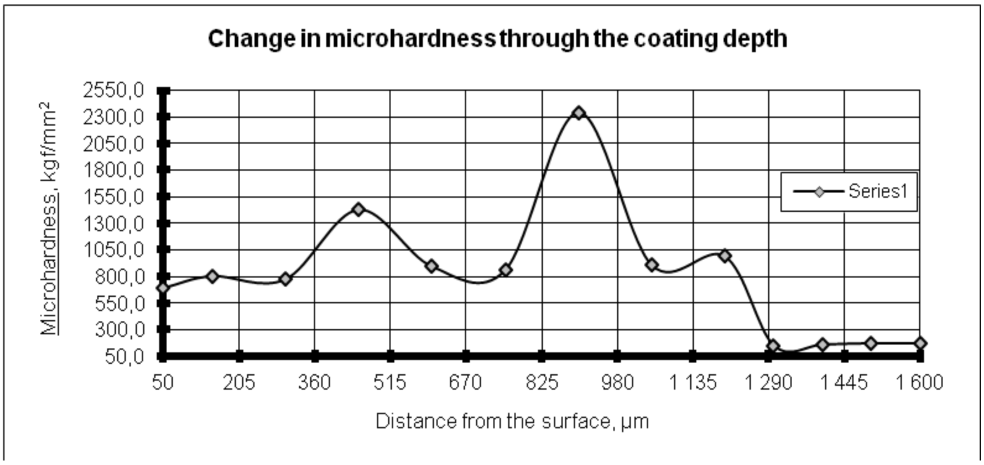


(a) ×200 (b) ×500

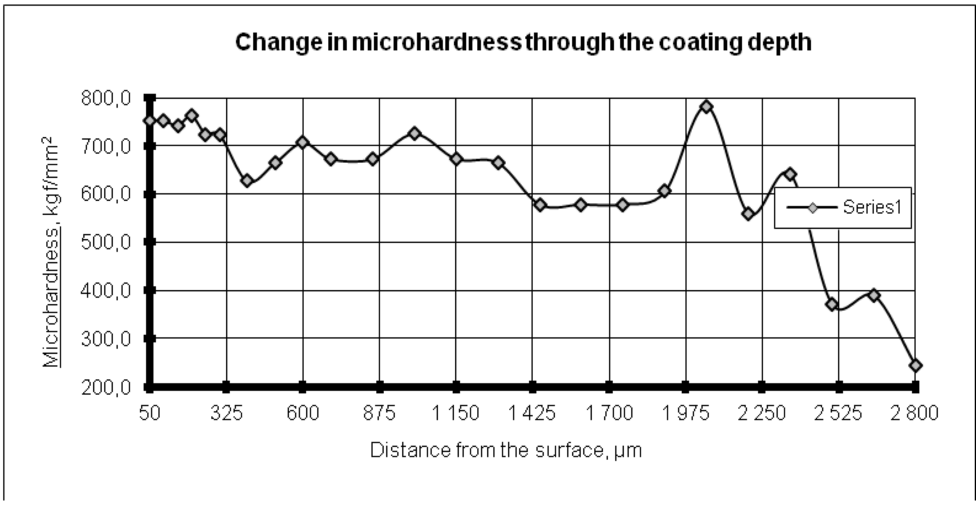
c) ×200 d) ×500

Fig. 4. Microstructure of sprayed coatings from the steels: (a, b) Sv-08G2S and (c, d) 40Kh13

The results of microhardness measurements of EAS coatings conducted on a microhardness tester “Micromet II” with a load of 100 g are presented in Fig. 5. Nitrogen saturation of the 40Kh13 steel coatings under the chosen conditions leads to the formation of a modified 150–200 μm thick surface layer (Fig. 4) with a microhardness of 6.5…7.0 MPa (Fig. 5).



(a)



(b)

Fig. 5. The microhardness distribution in EAS coatings from steel 40Kh13: the initial state; (b) after PIN treatment

The investigation for adhesion of EAS coatings showed that PIN can markedly increase the adhesion strength (Table 5). This increase is characteristic for coatings having porosity in the range of 5–14%; the further increase in porosity leads to decrease in adhesion.

The adhesion increase in the EAS coatings after PIN is due to the evolution of diffusion processes at the coating -substrate boundary, the recovery of oxides on the substrate surface, and the relaxation of internal stresses in the coating.

Table 5

The effect of porosity on the adhesion strength of EAS coatings (with no sublayer)

Material of coating	Adhesion strength, MPa, at different porosity, %					
	≤ 4	5–9	7–10	10–14	12–16	13–17
Sv-08G2S	38	49	61	53	40	36
40Kh13	37	43	55	47	43	35
40Kh13	41	55	67	63	50	39

The treatment of EAS coatings with PIN leads to a sharp increase in their wear resistance. Thus, the wear rate of coatings from steel 40Kh13 under dry friction decreases from 350 down to 19 μm/km. At the stage of steady friction, the wear rate of coatings after PIN treatment decreases to 5–6 μm/km. At the same time, the wear resistance of the modified layer from steel 40Kh13 is 1.6 times higher than that of cast steel 45 in the thermally hardened state (Fig. 6).

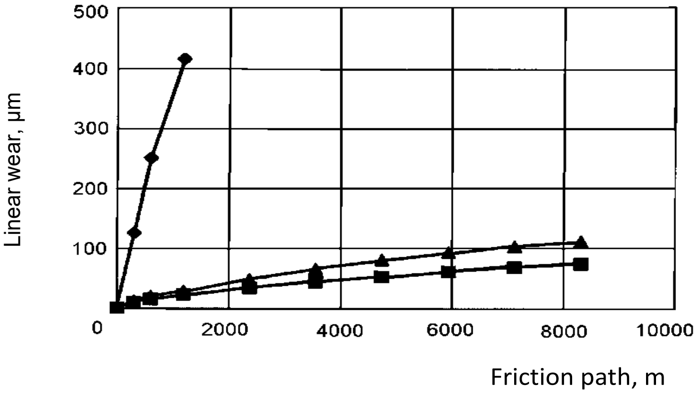
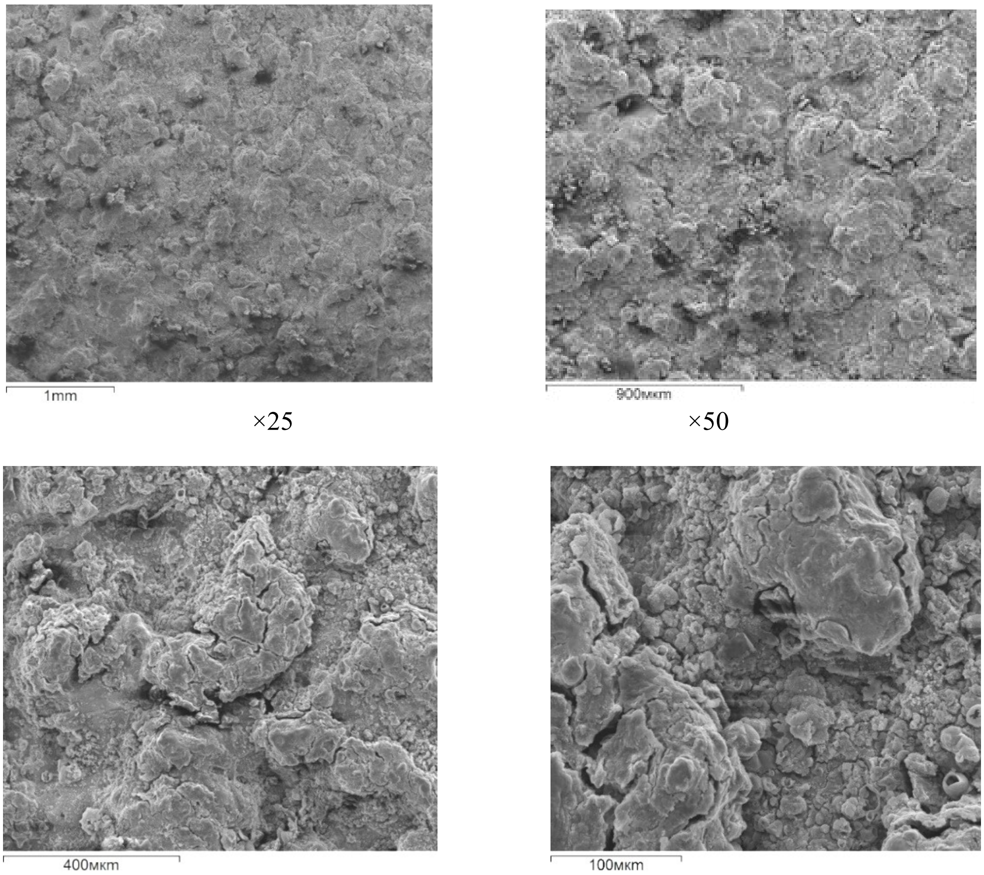


Fig. 6. Dependence of the linear wear on the dry friction path for coatings from steel Sv-08G2S: ■ coating Sv-08G2S + PIN; ▲ cast steel 45 (quenching and tempering); ♦ coating Sv-08G2S (initial state)

The findings of the surface topography examination for EAS coatings after PIN are presented in Fig. 7.



×25 ×50

×100 ×300

Fig. 7. Surface topography of EAS coatings after PIN

Conclusions

This study was devoted to developing and testing techniques for formation of hardened protective coatings on the basis of combination of EAS with subsequent PIN.

The possibility to improve the quality of EAS coatings from steels of martensitic (40Kh13, 95Kh18), ferritic (Sv-08G2S), and austenitic (Kh18N10T, 12Kh18N10T) grades thanks to subsequent ion-nitriding treatment has been evaluated.

The phase composition and microhardness of coatings obtained *via* spraying wires from ferritic, austenitic, and martensitic steels have been investigated. As a result of experimental studies of the influence of modifying effects of PIN on the physicomechanical properties of EAS steel coatings. It was established that in order to increase the efficiency of the modification process, porosity of coatings should be not more than 7%, which allows the formation of surface layers with a microhardness of 6.5 - 15.0 GPa and a thickness of 20-50 μm.

It was shown that the EAS method combined with subsequent PIN allows the formation of coatings with a surface layer with a hardness of 6.5 to 15.0 GPa and a thickness of 20 to 50 μm.

The wear resistance of EAS coatings from 40Kh13 and Kh18N10T steels after PIN increases by eight times. The amount of oxides in the coatings does not change after PIN treatment and favorably affects the performance of the coatings under the conditions of dry friction in air.

Saturation with nitrogen of EAS coatings from wire steel 40Kh13 leads to the formation of a diffusion 40-50 μm thick layer, the microhardness of which is 6.5 – 15 GPA. Herein the adhesion strength of the coatings increases by 1.6 times, and wear resistance does by 15–20 times.

It was shown that a duplex technology, combining EAS and PIN, allows one to obtain a significant effect concerning the bearing capacity, increase in hardness, adhesion strength, and wear resistance of the part-coating system along with reducing the probability of the part deformation. Discrete-pulse energy input at PIN increases the rates of heating and nitrogen diffusion by 2.5 times and shortens nitriding time by 2.6 times compared to the classical chemical heat treatment. The PIN process does not provide a uniform heating of the whole part, but only the surface layer of the required depth for hardening. PIN changes neither the shape and dimensions of the part, nor the roughness of its surface, so it can be used as a finishing treatment. The cyclical nature of the heating process makes it possible to reduce the power supply by 2.5 times.

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Лопата О.В., Лопата В.М., Качинська І.Р., Солових А.Є. Дослідження можливості підвищення якості електродугових покриттів шляхом обробки азотуванням

Ефективним способом вирішення завдань щодо зменшення витрат характеристик електродугових покриттів із дротяних матеріалів може виникнути утворення в них високоміцних поверхневих шарів за рахунок використання різних методів хіміко-термічної обробки. Були проведені дослідження з розробкою та апробуванням концепції формування зміцнювальних захисних покриттів на основі поєднання процесу електродного напилення з технологічною обробкою імпульсним іонним азотуванням. Показано, що дуплексна (комбінована) технологія, що поєднує електродугове напилення та азотування, дозволить отримати значний ефект за несучою здатністю, підвищення твердості, міцності зчеплення та зносостійкості системи деталь-покриття при зниженні ймовірності деформації деталі. Була проведена оцінка можливості підвищення якості електродугових покриттів з мартенситного сталей (40Х13, 95Х18), феритного (Св-08Г2С) і аустенітного (Х18Н10Т, 12Х18Н10Т) класів шляхом подальшої обробки азотуванням.

Ключові слова: комбіновані методи нанесення зносостійких покриттів, хіміко-термічна обробка, електродугове напилення, азотування,. зносо- та корозійну стійкість, зміцнюючи захисні покриття