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The influence of preliminary plasma treatment of the 09G2S steel surface on the formation of a coating as a result of hot galvanizing

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Abstract: In recent years, the range of silicon-containing steels subjected to hot galvanizing has been expanding. Alloying of steel with 0.5–1 % of silicon leads to the formation of a zinc coating of great thickness with a matte or multi-colored surface. This is associated with the changes in phase reactions between iron and zinc in the Fe–Zn–Si system. The development of ways to neutralize the negative influence of silicon on the formation of zinc coating is an urgent task. The purpose of the work is to study the influence of preliminary plasma cutting and plasma surface hardening of 09G2S (S355J2) steel on the thickness and structure of zinc coating formed on treated surfaces. It was found that after plasma cutting, the structure of the surface layer of steel is martensite, and after plasma surface hardening, it is martensite and ferrite. Analysis of the change in microhardness from the steel surface to the middle showed that the hardened layer depth is 400 µm. A zinc coating consisting of a δ-phase and a ζ-phase is formed on the surface of the steel without pretreatment. On the surface of the steel after plasma treatment, a zinc coating is formed characteristic of low-silicon steels and consisting of the δ-phase, ζ-phase, and η-phase. It was found that the thickness of the zinc coating on the surface after plasma cutting is two times less than on the untreated surface, and the reduction in the coating thickness occurs due to a decrease in the ζ-phase thickness. A hypothesis was suggested that the martensite formation on the steel surface leads to the disappearance of the ordered FeSi phase and changes the phase equilibrium in the Fe–Zn–Si system. Consequently, preliminary plasma treatment of the steel surface allows controlling the structure and thickness of the resulting zinc coating and is therefore recommended for introduction into the hot galvanizing process of silicon-containing steels.

Keywords: hot galvanizing; zinc coating; silicon-containing steels; Fe–Zn–Si; plasma treatment; surface hardening.

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INTRODUCTION

For the last two decades, an expansion in the range of steels subjected to hot galvanizing have been seen. It is associated with the need in the construction, and automotive industries for modern advanced high-strength steels (AHSS), including press hardening steel (PHS), austenitic plastic steels (Transformation Induced Plasticity, TRIP steels), dual-phase steels (DP steels), complex phase steels (CP steels), martensitic steels (MS), cold-deformable steels, etc. The unique complex of properties of these steels is high structural strength, low weight, and the ability to self-adapt to extreme external influences, which are ensured by thermomechanical processing, and an alloying system. Silicon is one of the main alloying elements that stabilize austenite [1; 2]. Silicon is also a cheap reinforcer of structural steels widely used for welded building structures, including 09G2S (S355J2) steel [3]. However, a high silicon content (more than 0.4 %) promotes the formation of a zinc coating of large thickness, 200–500 µm, on steel [4]. Such a thickness of the zinc coating leads to excessive consumption of zinc raw materials, and in some cases, to coating peeling, which is unacceptable. It has been found that this is associated with the influence of silicon on the processes of mutual diffusion of iron and zinc, during the coating formation [5–7].

It is known that zinc coatings produced by immersing steel in a melt, have in their structure, layers of intermetallic phases of the Fe–Zn system: δ, ζ, and η. These phases are well studied; they differ in structure, chemical composition and crystal lattice. The δ-phase layer is adjacent to the steel substrate, its thickness is uniform, and its structure is relatively compact. The next layer of the ζ-phase has a branched dendritic structure; the crystallites are elongated in the direction of heat removal from the base to the coating surface. When removing the product from the bath, almost pure zinc is formed on the surface – the η-phase [8].

When galvanizing steels with a silicon content of more than 0.4 %, the η-phase is absent, and the morphology of the ζ-phase changes, it becomes coarse-crystalline, and makes up about 90 % of the total coating thickness. In this case, the ζ-phase reaches the surface of the coating, giving it a matte appearance [9].

One of the options for controlling the growth of the zinc coating thickness is to control the galvanizing temperature, since it has a decisive influence on the coating thickness, structure, and phase composition [10]. The technology of high-temperature galvanizing (530–590 °C) is known, which makes it possible to produce coatings consisting mainly of the δ-phase – the densest phase providing a minimum coating thickness, including on silicon steels [11]. However, this technology requires the use of ceramic baths, since the steel ones have a shorter service life, and energy costs increase.

Another way to neutralize the negative influence of silicon on the galvanizing process, is to remove it from the steel surface by a special chemical pre-treatment, before hot galvanizing. It includes etching steel in complex solutions containing hydrofluoric and hydrochloric acids or ammonium, and sodium fluorides in various concentrations [12]. The disadvantage of this technology is the difficulty of recycling etching solutions.

Systems for alloying zinc melt with nickel, aluminum, bismuth, and tin have been developed, and implemented to control the structure, thickness and properties of coatings on silicon-containing steels [13]. For steels with a silicon content of up to 0.3 %, the application of zinc melt with nickel microadditives (0.05 %) has proven to be successful [14]. However, with a silicon content of more than 0.3 %, it was not possible to achieve a significant reduction in the coating thickness.

Before hot galvanizing, it is possible to apply preliminary metal coatings, such as iron, nickel, copper and/or their alloys, which form a diffusion barrier and prevent the formation of coating defects [15; 16].

The authors of this work found, that on the surfaces of steel parts after plasma and laser cutting, the coating thickness is significantly less than on other surfaces. Preliminary studies have shown that the insufficient thickness of the zinc coating on surfaces after plasma and laser cutting, depends not on surface defects, and the presence of scale, but on structural changes in the heat-affected zone. As a result of the impact of the torch during plasma cutting, the metal melts and quickly cools due to heat removal deep into the metal, therefore the near-surface structure is characterized by the presence of a martensitic component [17]. By plasma surface hardening, it is possible to obtain a surface martensitic layer both in the cut zone and on any machined surface [18]. The essence of the surface plasma hardening method is the rapid heating of the surface, by plasma jet exposure, and subsequent rapid cooling by removing heat into the bulk metal, which remains cold. Therefore, it is important to study in detail the influence of structural changes during plasma treatment of steel, on the formation of a zinc coating in order to develop new methods for controlling the zinc coating thickness.

The purpose of this research is to study the influence of preliminary plasma cutting and plasma surface hardening of 09G2S (S355J2) steel, on the zinc coating thickness and structure.

METHODS

For the study, samples of industrial hot-rolled 09G2S (S355J2) steel sheet in accordance with GOST 5520-79 with dimensions of 100×100×25 mm were selected. The chemical composition of the samples is determined on a Foundry-Master XPR optical emission analyzer and is shown in Table 1.

Samples were cut using a HyPerfomance 400 XD plasma cutter. Surface hardening of the samples was carried out using a UDGZ-200 manual plasma-hardening machine. The principle of its operation, is that the plasma flow heats the surface of the product very quickly, and due to heat removal from the surface into the depth of the product, the surface layer with a thickness of 1–2 mm is hardened. Hardening occurs in air without forced cooling with water or oil. The distance from the plasmatron to the surface of the product was 20 mm, the hardening current was 200 A, and the productivity was 110 cm/min.

Sections for studying the microstructure were made on a Remet LS 2 grinding and polishing machine. After hardening, the hardness of the samples was measured on a NOVOTEST TS-BRV stationary hardness tester using the Rockwell scale; measurements were taken from the machined side, and from the ends. The microstructure of the samples was studied using a Carl Zeiss Axio Vert 40 MAT digital trinocular inverted microscope. Then the samples were subjected to hot galvanizing, which included the following stages: degreasing in 20 % NaOH, etching in 10 % HCl, fluxing in $ZnCl_2$–NH_4Cl, drying, and immersion in zinc melt at a temperature of 450±3 °C; the time of holding in the melt is 2 min.

Microstructure studies and thickness measurements of the coating phase layers were carried out using a TESCAN VEGA SB scanning electron microscope. The microhardness of the subsurface layer of the samples was measured using a PMT-3 microhardness tester with a load of 20 g. It is important to note that the key factor in the formation of the structure, and properties of the heat-affected zone is the cooling rate. In our study, cooling occurred in air, which influenced the nature of the change in the microstructure. Air was chosen as a coolant, considering that the heating depth during surface hardening is small, so the heat is transferred into the sample thickness.

RESULTS

As a result of the analysis of the cross section of the sample after plasma hardening and the sample after plasma cutting, images of the microstructure were obtained, which are shown in Fig. 1 and 2.

In the microstructure of the sample after surface hardening (Fig. 1 a), a clear boundary between the surface structure, and the middle is visible – this is the depth of thermal influence, it amounts to about 950 μm. The near-surface layer is heterogeneous and has an incompletely hardened structure – martensite and ferrite (Fig. 1 b). The depth of the hardened layer is about 300 microns. The transition layer is represented in the form of a ferrite-pearlite structure (Fig. 1 c).

Table 1. *Chemical composition of 09G2S steel, %*
Таблица 1. *Химический состав стали 09Г2С, %*

Fe	C	Si	Mn	Ni	Cr	V	Cu
Base	0.137	0.608	1.670	0.015	0.038	0.012	0.009

In the microstructure of the sample, after plasma cutting (Fig. 2 a), there is a clear boundary between the surface structure, and the middle, the depth of thermal influence is about 600 µm. The near-surface layer is acicular martensite (Fig. 2 b). The hardened layer thickness is about 200 µm.

The depth of the hardened zone can be determined more accurately by analyzing the change in hardness, from the edge of the surface into the depth of the sample. Analysis of the graphs (Fig. 3) shows that hardness decreases from the edge to the middle. After plasma hardening, the hardness is uneven (martensite + ferrite). The maximum hardness value at the surface edge is 153 HV (martensite grain); the minimum hardness value is 123 HV (ferrite). A noticeable decrease in hardness occurs after a depth of 400 µm. The maximum value of steel hardness after plasma cutting at the edge of the surface was 173 HV. At a distance of 300–400 µm from the surface edge, the hardness decreases sharply and corresponds to the hardness of ferrite, i. e. this is the depth of the hardened zone.

The zinc coating on the samples without treatment has matte and multi-colored spots (Fig. 4 a), while on the sample after plasma treatment, the coating had a glossy shine (Fig. 4 b).

Studies of the zinc coating microstructure, obtained on surfaces without treatment and after plasma cutting and hardening (Fig. 5), showed that the phase structures of the coatings are different. On the surface without treatment, a zinc coating is formed, which has a structure characteristic

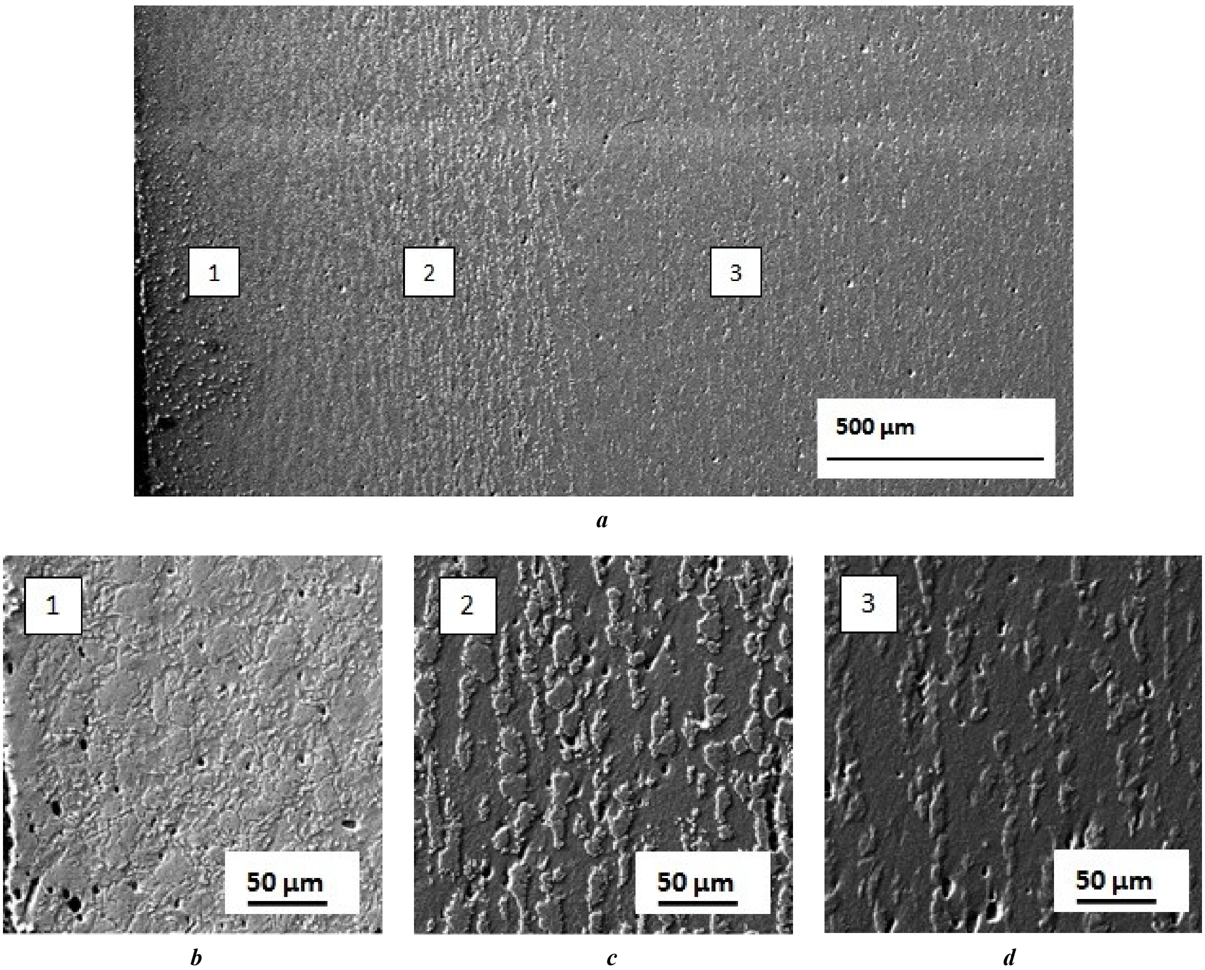

Fig. 1. *Microstructure of the sample after plasma hardening:*
a *– general view, ×100;* ***b*** *– surface layer, ×1000;* ***c*** *– transition layer, ×1000;* ***d*** *– middle, ×1000*
Рис. 1. *Микроструктура образца после плазменной закалки:*
a *– общий вид, ×100;* ***b*** *– поверхностный слой, ×1000;* ***c*** *– переходный слой, ×1000;* ***d*** *– середина, ×1000*

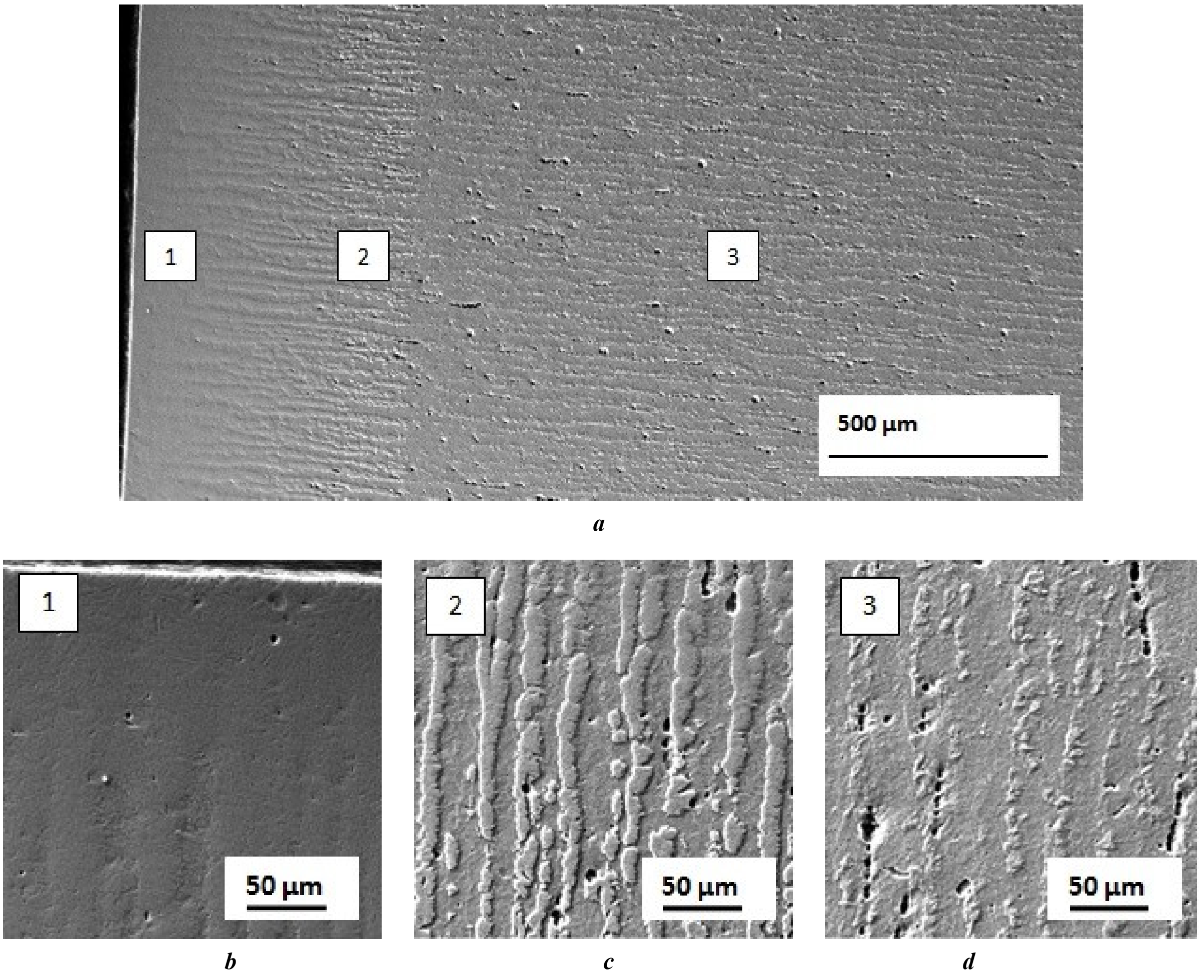

Fig. 2. *Microstructure of the sample after plasma cutting:*
a *– general view, ×100;* ***b*** *– surface layer, ×1000;* ***c*** *– transition layer, ×1000;* ***d*** *– middle, ×1000*
Рис. 2. *Микроструктура образца после плазменной резки:*
a *– общий вид, ×100;* ***b*** *– поверхностный слой, ×1000;* ***c*** *– переходный слой, ×1000;* ***d*** *– середина, ×1000*

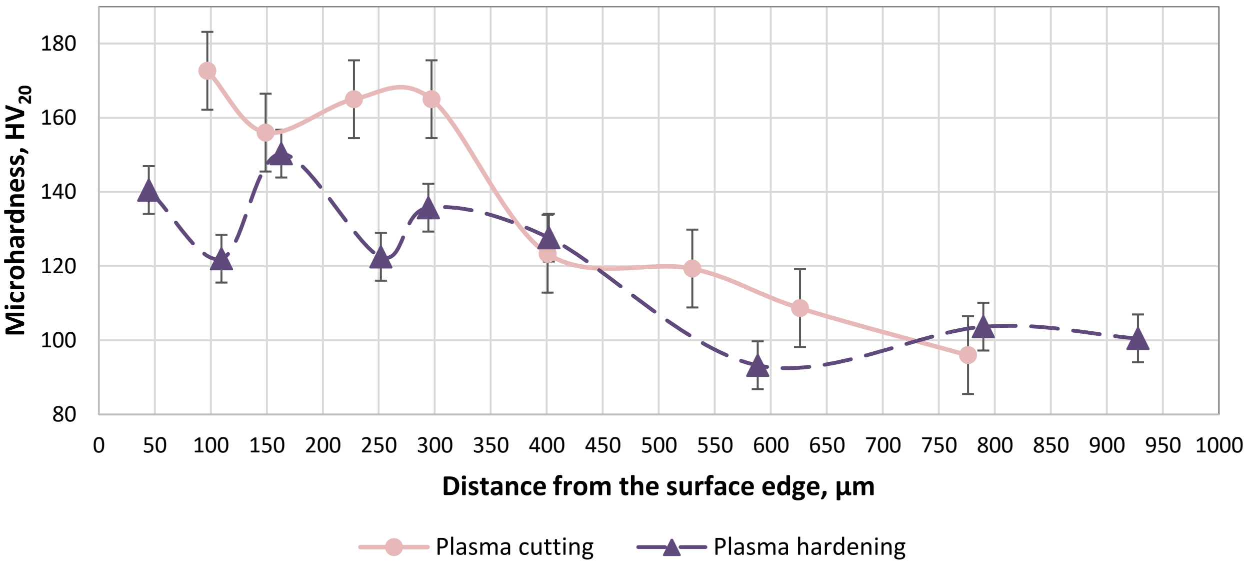

Fig. 3. *Change in microhardness from the edge of the treated surface to the middle*
Рис. 3. *Изменение микротвердости от края обработанной поверхности к середине*

for a coating on high-silicon steel: 80–90 % of the ζ-phase and a thin δ-phase layer, the η-phase is absent, which leads to a matte and multi-shaded coating surface (Fig. 5 a). After plasma treatment, all three main phases are observed in the microstructure of the zinc coating: the δ-phase is dense, columnar, the dendritic ζ-phase, and the η-phase is the coating zinc, which provides a glossy surface of the coating (Fig. 5 b, 5 c). This coating is typical for low-silicon steels. The thickness of the ζ-phase in the coating on the surface after plasma cutting is 30 % of the total coating thickness, and on the surface after plasma hardening – 50 % of the total coating thickness.

The analysis of the influence of preliminary plasma treatment, on the hardness of the sample surface, and the thickness of the forming zinc coating (Fig. 6), showed that the untreated sample with a ferrite-pearlite surface structure has the lowest hardness, and the greatest coating thickness, on average about 122 μm. After plasma hardening, the hardness of the ferrite-martensitic structure of the surface increased, and the thickness of the coating decreased by 29 % and amounted to about 87 μm. After plasma cutting, the hardness of the surface martensitic structure is maximum; the coating thickness decreased by 55 % relative to the untreated sample, and amounted to about 55 μm.

DISCUSSION

According to the Fe–Si equilibrium phase diagram, at low concentrations of silicon in iron, there are regions of the α-phase, which is a disordered solid solution of the substitution of iron by silicon in the body-centered cubic (bcc) lattice, and two ordered α1 (Fe_3Si) and α2 (FeSi) phases [19].

When silicon-containing steel, interacts with molten zinc, phase reactions are described by the Fe–Zn–Si ternary system. Researchers [20] showed that silicon present in steel in an amount of 0.5–1 %, forms the FeSi phase and shifts the equilibrium to the three-phase region: ζ-$FeZn_{13}$ – FeSi – liquid Zn. The presence of the liquid phase accelerates the growth of the ζ-$FeZn_{13}$ intermetallic layer.

As a result of the studies, it was found that the thickness and structure of the zinc coating, formed on 09G2S high-silicon steel, depends on the near-surface structure of the steel. If the structure is equilibrium, ferrite-pearlite, as in samples without heat treatment, then a thick zinc coating is formed with a well-developed ζ-phase, which is characteristic of high-silicon steel.

If the surface structure of the steel is martensite or martensite + ferrite, then the ζ-phase thickness decreases by 2 times, and a surface η-phase appears in the coating structure, which is typical for low-silicon steel. In this case, the near-surface structure of complete hardening (martensite), inhibits the growth of the coating ζ-phase more strongly than the incomplete hardening structure (martensite + ferrite).

One can assume that the martensite formation leads to the disappearance of the ordered FeSi phase. The martensite crystal lattice is greatly distorted compared to the ferrite lattice, and instead of cubic, it takes on a tetragonal shape. In the absence of the FeSi phase, the formation of a zinc coating occurs according to the Fe–Zn binary system, and phases specific to low-silicon steels are observed in the coating. It can be considered that the near-surface martensitic structure, is a diffusion barrier for silicon located in the steel, and influencing the formation and morphology of the zinc coating ζ-phase.

Thus, preliminary plasma treatment of the surface allows controlling the structure, and thickness of the zinc coating formed on this surface. In further research, it is planned to find the time of immersion of steel in the melt, during which the effect of the martensitic layer as a diffusion barrier is retained.

a ***b***

Fig. 4. *Coating surface after hot galvanizing:*
a *– without treatment;* ***b*** *– after plasma hardening*
Рис. 4. *Поверхность покрытия после горячего цинкования:*
a *– без обработки;* ***b*** *– после плазменной закалки*

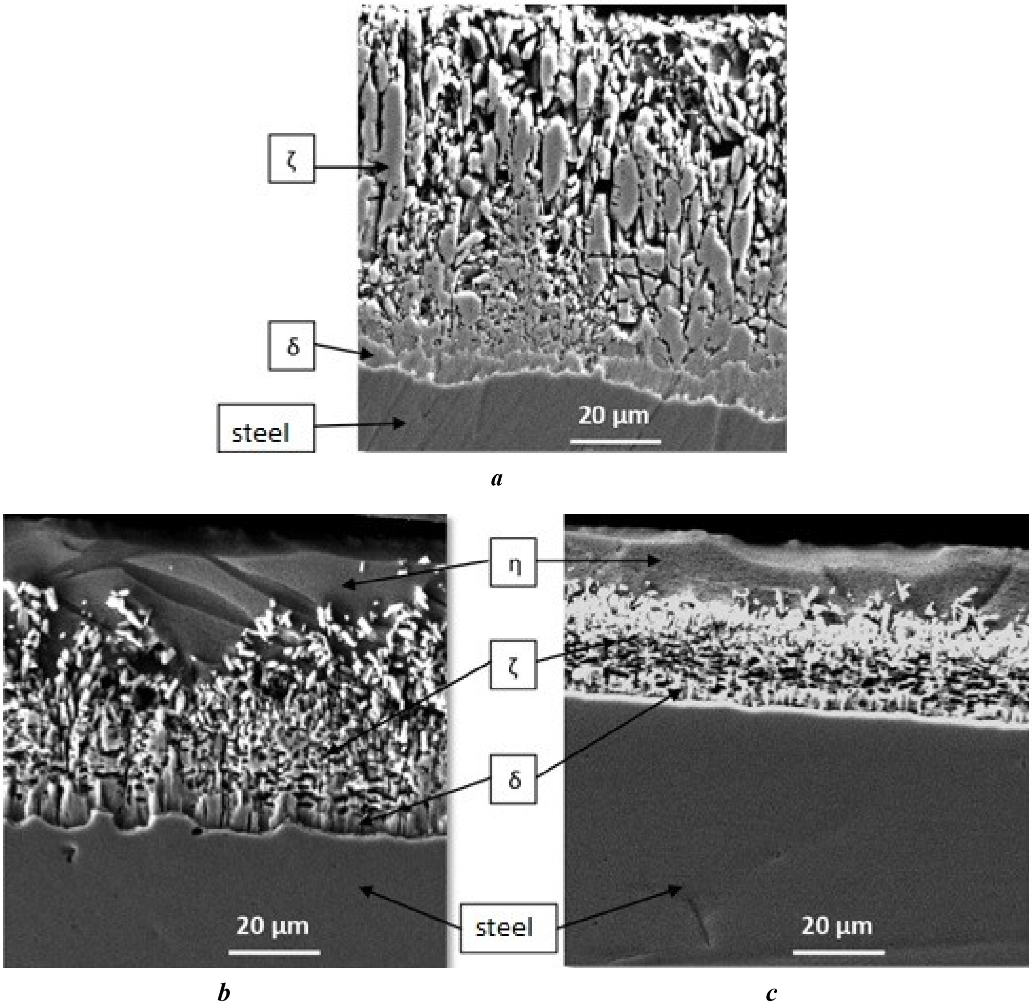

Fig. 5. *Microstructure of the sample zinc coating, ×2000:*
a *– without treatment;* ***b*** *– after plasma hardening;* ***c*** *– after plasma cutting*
Рис. 5. *Микроструктура цинкового покрытия образца, ×2000:*
a *– без обработки;* ***b*** *– после плазменной закалки;* ***c*** *– после плазменной резки*

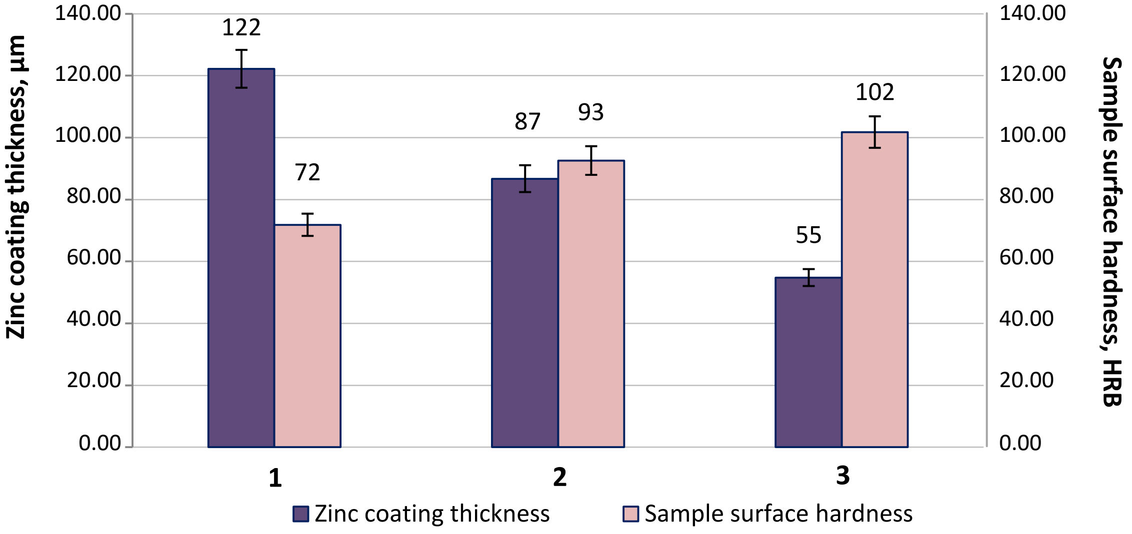

Fig. 6. *Diagram of the influence of treatment on the coating thickness and base hardness:*
1 – without treatment; 2 – plasma hardening; 3 – plasma cutting
Рис. 6. *Диаграмма влияния обработки на толщину покрытия и твердость основы:*
1 – без обработки; 2 – плазменная закалка; 3 – плазменная резка

CONCLUSIONS

The structure of individual sections of the heat-affected zone was determined, and their microstructure was studied. A completely hardened structure (martensite) was found on the surface after plasma cutting; an incompletely hardened structure (ferrite and martensite) was found on the surface after plasma hardening.

The maximum hardness of the sample surface after plasma cutting is 172 HV (increased by 42 %), and after plasma surface hardening – 153 HV (increased by 29 %). The hardened zone depth is 400 µm.

After plasma treatment, a coating with the pure zinc η-phase is formed on the surface, which gives the coating a glossy shine. The thickness of the zinc coating on the surface after plasma hardening is 29 % less, and on the surface after plasma cutting is 55 % less than on the untreated surface. The reduction in coating thickness occurs due to a decrease in the ζ-phase thickness.

A hypothesis was suggested, that the martensite formation on the steel surface leads to the ordered FeSi phase disappearance, and changes the phase equilibrium in the Fe–Zn–Si system. The zinc coating is formed according to the Fe–Zn binary system. Thus, the hardened layer serves as a diffusion barrier during the formation of the iron-zinc coating layers.

Preliminary plasma treatment of the 09G2S steel surface, leads to a reduction in the zinc consumption for the formation of a protective coating, and the formation of a glossy surface, therefore, the authors recommend to implement this method in the process of hot galvanizing of this steel grade.

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Влияние предварительной плазменной обработки поверхности стали 09Г2С на формирование покрытия в результате горячего цинкования

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Аннотация: В последние годы расширяется ассортимент кремнийсодержащих сталей, подвергаемых горячему цинкованию. Легирование стали 0,5–1 % кремния приводит к образованию цинкового покрытия большой толщины с матовой или разнотонной поверхностью. Это связано с изменением фазовых реакций между железом и цинком в системе Fe–Zn–Si. Актуальной задачей является разработка способов нейтрализации негативного влияния кремния на формирование цинкового покрытия. Цель работы – изучение влияния предварительной плазменной резки и плазменной поверхностной закалки стали 09Г2С (S355J2) на толщину и структуру цинкового покрытия, образующегося на обработанных поверхностях. Установлено, что после плазменной резки структура приповерхностного слоя стали представляет собой мартенсит, а после плазменной поверхностной закалки – мартенсит и феррит. Анализ изменения микротвердости от поверхности стали к середине показал, что глубина закаленного слоя составляет 400 мкм. На поверхности стали без предварительной обработки формируется цинковое покрытие, состоящее из δ-фазы и ζ-фазы. На поверхности стали после плазменной обработки формируется цинковое покрытие, характерное для малокремнистых сталей и состоящее из δ-фазы, ζ-фазы и η-фазы. Установлено, что толщина цинкового покрытия на поверхности после плазменной резки в два раза меньше, чем на необработанной поверхности, причем сокращение толщины покрытия происходит за счет уменьшения толщины ζ-фазы. Выдвинута гипотеза, что образование на поверхности стали мартенсита приводит к исчезновению упорядоченной фазы FeSi и изменяет фазовое равновесие в системе Fe–Zn–Si. Следовательно, предварительная плазменная обработка поверхности стали позволяет управлять структурой и толщиной образующегося цинкового покрытия и поэтому рекомендуется для внедрения в процесс горячего цинкования кремнийсодержащих сталей.

Ключевые слова: горячее цинкование; цинковое покрытие; кремнийсодержащие стали; Fe–Zn–Si; плазменная обработка; поверхностная закалка.

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