Article
https://doi.org/10.31489/2025PH1/68-76
UDC 621.7.02

Received: 28.11.2024
Accepted: 28.01.2025

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The Analysis of Structure Change and Tribomechanical Properties of Alloyed Steel Surfaces Modified by Diffusion Electrolyte-Plasma Boriding Method

Nowadays, one of the key requirements in mechanical engineering when manufacturing parts from constructional steels is the hardness and wear resistance parameters. One of the relevant solutions to this issue could be diffusion-electrolytic-plasma boriding, as the steel surface is enriched with boron elements during treatment, while the core of the part remains in its original state. This study addresses the technological capabilities of the diffusion-electrolytic-plasma boriding method for steels. The steel 30CrMnSiA was treated on a diffusion-electrolytic-plasma boriding setup. The treatment duration was 5 and 7 minutes, using a 15 % sodium carbonate (Na_2CO_3) and 20 % borax ($Na_2B_4O_7$) aqueous solution as the electrolyte. It was established that the cross-sectional structure of the steel after diffusion-electrolytic-plasma boriding is characterized by zoning, with the formation of a modified layer approximately 650 µm thick. As a result of diffusion-electrolytic-plasma boriding, the microhardness of 30CrMnSiA steel is enhanced by 2.5 to 3 times in comparison to its original state, due to the formation of hardening phases.

Keywords: diffusion, electrolyte, plasma, boriding, structure, surface modification, surface, borax

Introduction

Traditional steel processing methods have focused on thermal processes such as heating and cooling, which have been used for a long time. These techniques require heating the metal in a furnace followed by cooling it in air, water, or oil [1]. However, these processes can be expensive due to the need for specialized equipment and extended processing times, and they often require large, cumbersome machinery [2]. Recently, an innovative method of processing steel parts, electrolyte-plasma heat treatment, has emerged that overcomes many of the limitations of traditional methods [3]. This method can significantly improve the physical and mechanical properties of steel in a short time — only a few seconds, unlike traditional methods, which can take hours and days. Electrolyte-plasma hardening is an intricate process that merges physical metallurgy with electrochemical technologies. It involves heating the sample in cathodic mode, enabling simultaneous phase transformation and deformation [4].

To improve the strength characteristics of structural and tool steels, a thermocyclic approach known as thermocyclic electrolytic-plasma treatment is used within the framework of electrolytic-plasma treatment [5]. Unlike other types of heat treatment, this method involves multiple repetitions of the heating and cooling cycle, which allows to significantly improve the quality of the material and achieve properties that cannot be obtained with a single heat treatment step. The changes from cycle to cycle are most commonly attributed to plastic deformation. The efficiency of thermocyclic electrolytic-plasma treatment is mainly influenced by the interactions between various phases and the structural transformations in alloys caused by microplastic deformation and the subsequent recrystallization process. Microplastic deformation boosts the strength of steel, while recrystallization enhances its ductility [6].

The effectiveness of electrolyte-plasma thermocyclic treatment is significantly influenced by factors such as cycle temperatures, the number of cycles, and the rates of heating and cooling [7]. The range of methods for thermocyclic electrolyte-plasma treatment, which differ in terms of their operational principles (including complete, partial, or no phase transformations) and energy requirements, which can vary by a factor of 20 to 50, offers potential for optimizing the process [8]. This highlights the necessity for developing and implementing more efficient technologies for the hardening of structural and tool steels to enhance product quality, operational lifespan, and reduce energy consumption, which is crucial for economic considerations.

Traditional furnace heat treatment methods face a number of restrictions, such as the risk of deformation, warping and cracking, long heating (1 to 20 hours) and cooling cycles, as well as limited automation and other difficulties.

In mechanical engineering, multi-component chemical heat treatment (CHT) is commonly applied to improve the surface characteristics of steels and alloys. This process involves either simultaneous or sequential diffusion of various chemical elements into the surface layers [9]. Techniques like nitriding and carbonitriding, which are part of CHT, significantly enhance the wear resistance, corrosion resistance, and other performance attributes of machine parts. However, the full understanding of how nitrided and carbonitrided layers form, with a combination of desirable properties such as high wear and corrosion resistance, is still incomplete [10]. Conventional chemical-thermal treatments, like nitriding in a gas environment and carburizing with solid carburizers, have limitations, including challenges in controlling the diffusion-active medium, the need for expensive equipment, and the necessity of multiple heat treatment cycles to achieve the final desired properties of the product.

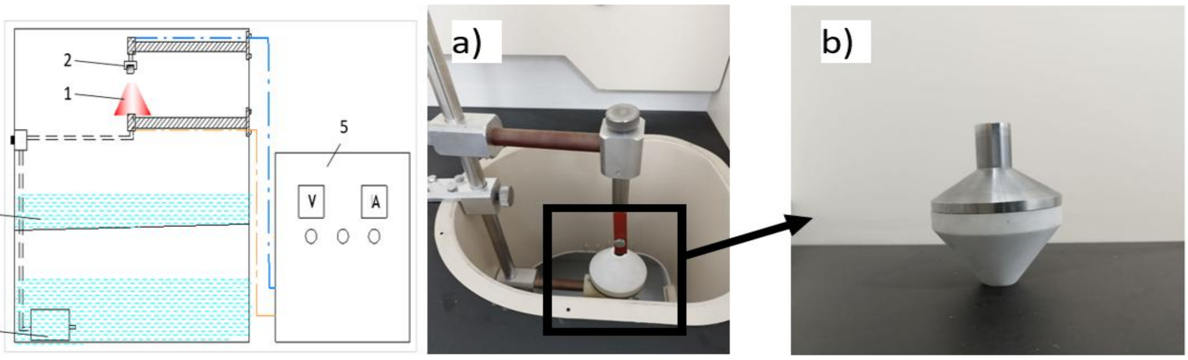
A promising approach to enhancing the service life and performance of steel components is chemical-thermal treatment with electrolyte-plasma action (diffusion saturation). This method is used to introduce light elements such as nitrogen, boron, and carbon into the surface of materials [11]. Compared to traditional surface modification techniques, this technology offers several advantages, including faster processing times, lower costs, and the ability to combine diffusion saturation with hardening in a single technological process. The results of studies conducted by various scientific groups confirm the significant potential of electrolyte-plasma treatment of metals and alloys, but the tribological characteristic of this method remains insufficiently studied [12]. It is important to conduct a more detailed analysis of the wear mechanism in steel products following electrolytic-plasma treatment and examine it under a variety of conditions.

Due to the above, the aim of this paper is to analyze the changes in the structure and tribomechanical surface properties of alloyed steel before and after diffusion electrolyte-plasma boriding.

Materials and methods

In this research work, samples from 30CrMnSiA alloy steel with the size of 33×30×9 mm were selected as research objects for diffusion-electrolyte-plasma boriding (DEPB). Before the experiments, the samples were pre-treated with grinding paper with P2000 grit.

Figure 1 shows a general view of an image of the modernized diffusion-electrolyte-plasma boriding (DEPB) unit.

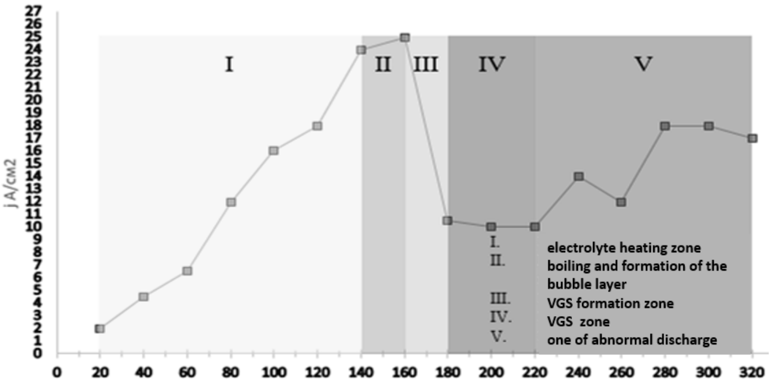


1 — anode; *2* — sample; *3* — electrolyte bath; *4* — pump; *5* — power supply; *a* — sample holder; *b* — nozzle

Figure 1. Image of the unit for diffusion-electrolyte-plasma boriding

The unit includes a high-power rectifier that provides direct current with a maximum output of 360 V and 100 A. The electrodes are composed of a cathode (sample) and an anode, which is shaped like a circular plate with perforations to ensure the even distribution of the electrolyte. The electrolyte used is an aqueous solution containing soda ash (Na_2CO_3) and borax ($Na_2B_4O_7$) in certain proportions, which is considered optimal for the formation of stable plasma [13].

The key factor in the process (DEPB) is the potential difference between the anode and cathode [14]. To determine the value of the potential difference of this process, the volt-ampere characteristics (VAC) of the anode and cathode were plotted (Fig. 2). The first region (I) region of linear dependence of current and voltage ($U = 0–140$ V i.e. (zone of classical electrolysis) [15]. The second region (II) of electrolyte boiling followed by vapor formation is characterized by a slow increase in current, which is explained by heating of the active electrode. Region (III–IV) is the zone of formation of a stable vapor-gas shell (VGS) around the cathode, which explains the sharp decrease in current. At further voltage increase, the breakdown of the VGS and the transition of the system to an unstable state occur [16].



U, B	20	40	60	80	100	120	140	160	**180**	**200**	**220**	240	260	280	300	320
I, A	2	4.5	6.5	12	16	18	24	25	**10.5**	**10**	**10**	14	12	18	18	17

Figure 2. VAC plot of the DEPB process [17]

Experimental studies were carried out in the Research Center “Surface Engineering and Tribology” of S. Amanzholov East Kazakhstan University (Ust-Kamenogorsk, Kazakhstan). The phase composition of materials was determined using X'PertPro X-ray diffractometer (Philips, Netherlands). To reveal the microstructure of the samples we applied chemical etching using 4 % solution of nitric acid (HNO_3) in ethyl alcohol. The microstructure was studied using an Olympus BX53M optical metallographic microscope. The depth hardness was determined using a Vickers microhardness tester (Metolab 502, RF) equipped with a diamond indenter and a load cell up to 1000 g.

The values of coefficient of friction were determined on TRB3 tribometer, where rotation speed was 2 cm per second, radius of the track $R = 2$ mm; vertical load: 6 N; a 100Cr6 ball of 6 mm diameter was selected as a counterbody.

Results and Discussion

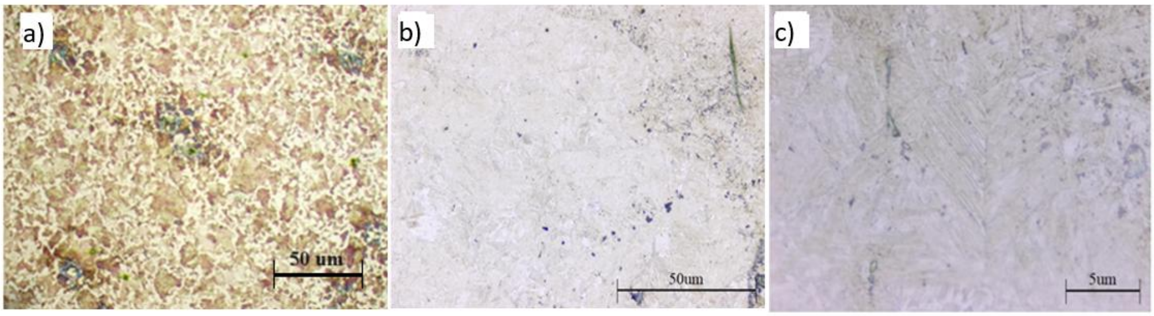
The samples underwent treatment for 5 minutes (№ 1) and 7 minutes (№ 2) with a DC current applied at 290 V, which corresponds to a temperature of 1000 °C. Thermocouples placed in predrilled holes on the surface of the samples were used to monitor their temperature. The experiments were performed in cathode mode, and the key process parameters are presented in Table 1.

Table 1

Table of DEPB modes

	U, V	T, °C	t, min	Electrolyte composition
Sample № 1	290	1000	5	65 % distilled water, 15 % sodium carbonate (Na_2CO_3) and 20 % borax ($Na_2B_4O_7$)
Sample № 2			7	

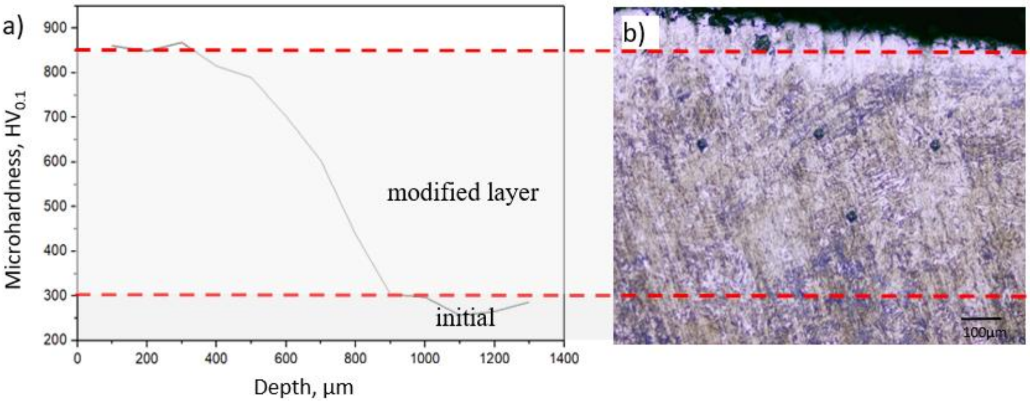
Figure 3 shows the microstructure of steel 30CrMnSiA before and after DEPB, the main structure of steel is martensite with strengthening structure of boride (light needle-like) and cementite (dark along the grain boundaries of martensite). The study of steels with martensitic structure revealed their high hardness [18].



a — microstructure of steel in initial state, magnified in ×50; *b* — microstructure of sample № 1 after DEPB, magnified in ×50; *c* — microstructure of sample № 2 after DEPB, magnified in ×100

Figure 3. Microstructure of steel 30CrMnSiA before and after DEPB

It has been determined that the cross-sectional structure of 30CrMnSiA steel after DEPB has a zonal characteristic: 1 zone of the modified layer, which consists of boride and thermal influence zones, and 2 zone of the matrix — base of the treated material. The analysis of hardness distribution along the thickness of the modified layer after DEPB is particularly interesting. The study of microhardness along the depth showed the presence of a harder surface layer and a less hard layer under it, the extent of which is about 600 μm. The graph in Figure 4 shows that the microhardness gradually decreases from the surface to the middle of the specimen.



a — microhardness of hardened layers; *b* — microstructure of modified layer

Figure 4. Microhardness of 30CrMnSiA steel by thickness after DEPB (№ 2)

In the near-surface layers microhardness reaches an average of 960 $HV_{0.1}$, then with a smooth transition decreases to an average value of 613 $HV_{0.1}$ in the heat affected zone. In the initial state, the microhardness of the steel is 280±10.4 $HV_{0.1}$.

After DEPB steel 30CrMnSiA underwent significant qualitative and quantitative changes in the steel structure, and, as shown by the studies, the fine structure of steel and phase composition in the near-surface zone of the modified sample and in the transition zone (at a depth of ~650 μm) was significantly different.

As a result of the conducted research, it was found that the material of the near-surface layer is multiphase. However, the main component remains α′-phase (α′-Fe). It should be noted that no ferrite and pearlite grains were found on the sample surface after DEPB (Fig. 4, *b*).

Figure 5 shows the XRD-results of 30CrMnSiA steel before (Fig. 5 *a*) and after (Fig. 5 *b, c*) DEPB. In the initial state, 30CrMnSiA steel consists mainly of α-Fe phase with face-centered cubic lattice (FCC). After the DEPB process, cementite (Fe_3C), boride (Fe_2B), iron oxide (Fe_2O_3) and martensite (α′-Fe) phases are found in 30CrMnSiA steel. Martensite is formed in the modified steel layer as a result of rapid cooling during the DEPB process. The results of X-ray phase analysis are in agreement with the results of studying the microstructure of 30CrMnSiAsteel after DEPB.

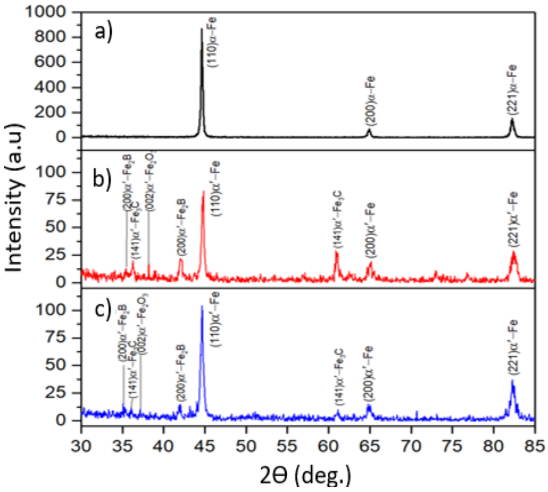


Figure 5. XRD of 30CrMnSiA steel before DEPB (*a*) and after DEPB № 1 (samples *b*) and № 2 (*c*)

During DEPB, 30CrMnSiA steel is exposed to high temperatures produced by electric current. These elevated temperatures may cause the austenite (the stable crystalline form of iron) to transform into more stable phases like martensite and cementite. The structure and properties after DEPB depend primarily on the temperature and duration of the interaction of the electrolyte with the sample surface and the composition of the electrolyte.

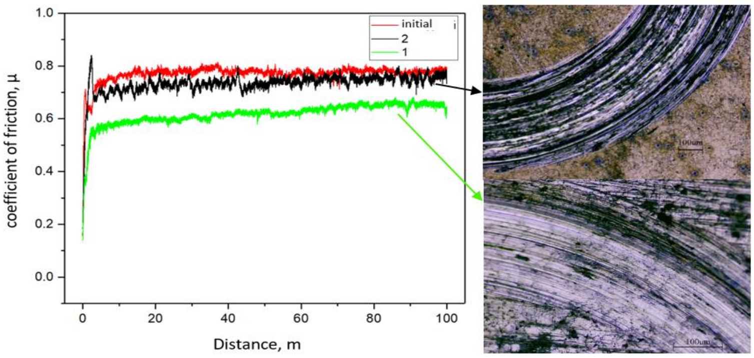


Figure 6: Histogram of sample friction readings and wear trace after tribological testing

The results of tribological tests of boronized steel showed (Fig. 6) that after DEPB, 30CrMnSiA steel has a lower coefficient of friction compared to the original sample. Thus, we can assume that after DEPB the wear resistance of 30CrMnSiA steel increased compared to the original sample.

Table 2

Correlation table of experimental data

	Phase	Microhardness, $HV_{0.1}$	Coefficient of friction, μ
Initial	Ferrite, perlite	280±10.4	0.89
Sample № 1 (5 min)	Cementite, boride, iron oxide and martensite	639.34±16.3	0.68
Sample № 2 (7 min)	Cementite, boride, iron oxide and martensite	659.76±16.7	0.76

Table 2 shows the correlation experimental data that were carried out during the study. From the Table 2, we can see that after DEPB, 30CrMnSiA steel underwent changes affecting not only its microhardness but also its phase structures. According to the XRD results, martensite, cementite, boride and iron oxide phases are formed on the surface of 30CrMnSiA steel, but the martensite phase (α′-Fe) is the dominant phase in the steel structure following boriding. Also, after DEPB the wear resistance of the samples changed for the better. In the histogram in Figure 6 we see that the friction coefficient of the samples after DEPB is less than that of the original sample.

Conclusions

In this research work, diffusion electrolyte-plasma boriding of 30CrMnSiA steel has been investigated. According to the results, the diffusion electrolyte plasma boriding (DEPB) method can be considered as an effective method for steel modification, which allows different types of treatment depending on the conditions and choice of electrolyte. The DEPB method demonstrates excellent effectiveness in the surface treatment of steels and offers considerable benefits over traditional heat treatment techniques, including energy efficiency, shorter treatment times, and improved environmental safety [19].

It was found that the structure of the cross-section of 30CrMnSiA steel after DEPB is conditionally divided into 3 zones: boride layer, heat affected zone, which together will form a modified layer and the base of the treated material. The structure of the steel cross-section after DEPB is characterized by zonality, with the formation of a modified layer with a thickness of about 650 μm, containing different phases: α′-Fe-martensite, Fe_3C-cementite. The microhardness of 30CrMnSiA steel after DEPB increases 2.5-3 times compared to the initial state due to the formation of hardening phases.

To improve the DEPB plant, additional modules such as electrolyte plasma polishing, which is a finishing module, were retrofitted. The source for EPB was reassembled. The optimum mode for DEPB was selected, including parameters, electrolyte composition, VAC. A nozzle (Fig. 3, *b*) was assembled specially for the modernized DEPB unit.

Acknowledgment

This research is funded by the Science Committee of the Ministry of Science and Higher Education of the Republic of Kazakhstan (Grant No. AP19680473).

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Диффузиялық электролиттік-плазмалық борлау әдісімен легірленген болаттардың модификацияланған беттерінің құрылымы мен трибомеханикалық қасиеттерінің өзгеруін талдау

Қазіргі уақытта конструкциялық болаттардан бөлшектер жасау кезінде машина жасау саласындағы ең өзекті талаптардың бірі қаттылық пен тозуға төзімділік параметрлерінің болуы. Бұл мәселеге қатысты тиімді шешімдердің бірі диффузиялық-электролиттік-плазмалық борлау әдісі болуы мүмкін, себебі өңдеу барысында болаттың беті бор элементтерімен қанығады, ал бөлшектің өзегі бастапқы күйінде қалады. Бұл жұмыста болаттарды диффузиялық-электролиттік-плазмалық борлау әдісінің технологиялық мүмкіндіктері қарастырылған. 30ХГСА болатты өңдеу диффузиялық-электролиттік-плазмалық борлау қондырғысында жүргізілді. Өңдеу уақыты 5 және 7 минут болды, ал электролит ретінде 15 % кальцинделген сода (Na_2CO_3) және 20 % бураның ($Na_2B_4O_7$) судағы ерітіндісі қолданылды. Анықталғандай, болаттың көлденең қимасының құрылымы диффузиялық-электролиттік-плазмалық борлаудан кейін зоналдық сипатқа ие, ал модификацияланған қабаттың қалыңдығы шамамен 650 мкм. Диффузиялық-электролиттік-плазмалық борлау процесінің нәтижесінде 30ХГСА болатының микроқаттылығы қатайту фазаларының түзілуіне байланысты бастапқы күйімен салыстырғанда 2,5–3 есе артады.

Кілт сөздер: диффузия, электролит, плазма, борлау, құрылым, бетті модификациялау, беті, бура

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Анализ изменения структуры и трибомеханических свойств модифицированных поверхностей легированных сталей методом диффузионного электролитно-плазменного борирования

В настоящее время одним из актуальных требований в сфере машинострения при изготовлении деталей из конструкционных сталей, являются параметры твердости и износостойкости. Одним из решений этой проблемы может стать диффузионно-электролитно-плазменное борирование. При такой обработке поверхность стальной детали насышается бором, в то время как сердцевина остается в исходном состоянии. В данной работе рассмотрены вопросы технологических возможностей способа диффузионно-электролитно-плазменного борирования сталей. Была проведена обработка стали 30ХГСА на установке диффузионно-электролитно-плазменного борирования. Продолжительность обработки составляла 5 и 7 минут при использовании в качестве электролита водного раствора 15 % кальцинированной соды (Na_2CO_3) и 20 % буры ($Na_2B_4O_7$). Установлено, что после диффузионно-электролитно-плазменного борирования, структура поперечного сечения стали характеризуется зональностью, с образованием модифицированного слоя толщиной около 650 мкм. В результате образования упрочняющих фаз после диффузионно-электролитно-плазменного борирования, микротвердость стали 30ХГСА увеличивается в 2,5–3 раза по сравнению с исходным состоянием.

Ключевые слова: диффузия, электролит, плазменное, борирование, структура, модификация поверхности, поверхность, бура

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