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STUDY OF THE DIFFUSION ELECTROLYTE PLASMA TREATMENT EFFECT ON THE TRIBOLOGICAL CHARACTERISTICS OF 40Kh STEEL

Abstract: This article studies the effects of diffusion electrolytic plasma boriding on the structure and tribological characteristics of 40Kh steel. X-ray diffraction and scanning microscopy were used to investigate the structural and phase composition, while the mechanical and tribological properties of the steel were measured using a hardness tester and tribometer. The characteristics of 40Kh steel in its initial state and after diffusion electrolytic plasma treatment followed by oxide removal by electrochemical polishing were compared. After diffusion electrolytic-plasma treatment, FeB and Fe₂B boride phases were detected on X-ray diffractograms, proving the diffusion process. A 5 times increase in microhardness and a 3 times decrease in wear intensity were also determined, depending on the initial state. It was established that the morphology of the structure after boriding has a zonality consisting of three zones: the borided layer, the diffusion zone, and the heat-affected zone. The research data indicates the potential of using diffusion electrolytic-plasma treatment in mechanical engineering and the processing of parts for the mining industry.

Key words: diffusion electrolytic plasma treatment, boride, electrolyte, borax, microhardness.

Introduction

Improving the reliability and service life of mining and metallurgical equipment components requires the implementation of effective surface hardening technologies. In highly abrasive, corrosive and thermally stressed environments, wear and surface degradation are among the main causes of equipment failure. The most effective way to increase the wear and corrosion resistance of structural steels remains diffusion boriding, which allows the formation of superhard layers of FeB and Fe₂B phases with a hardness of up to 22 GPa and high chemical resistance [1]. In practice, various boronising methods are used: in powder mixtures, melts, gas phase, as well as electrolytic and plasma boron saturation. However, traditional approaches (in particular, thermal diffusion boronizing in powder mixtures) require long processing times (4-8 hours) and high temperatures

(850-1000 °C) [2], which limits their application to complex shapes, large structures, and parts with precise tolerances. Plasma boronising in a glow discharge, laser and electron beam saturation allow for a reduction in temperature and time parameters, but are characterised by the complexity of the equipment, the need for a vacuum environment, the toxicity of the reaction components (e.g. BCl_3 or B_2H_6) [3], and high cost. Electrolytic plasma boriding (EPB) is a promising surface modification method that combines ease of implementation, high process speed (5-30 minutes) and the ability to treat complex shapes. The process is carried out in aqueous solutions at a voltage of 150–350 V and the formation of a vapour-plasma shell, in which intensive heat and mass transfer and electrochemical processes take place. The processing temperature is 500-800 °C, depending on the voltage and electrolyte parameters [4]. The features of EPB include the formation of a dense boride layer with a microhardness of up to 1600-2000 HV, improved adhesion due to the gradient nature of the layer, the possibility of combining with further heat treatment in the same electrolyser, and the absence of toxic and expensive reagents. However, issues remain unresolved regarding the uniformity of layers on large parts, the stability of the layer morphology in areas of intense discharge, and the need for precise adjustment of electrolyte compositions for different steel grades [5].

Boriding methods using electrolytic plasma treatment, like other types of diffusion saturation under similar conditions, can be classified according to several criteria. One of the key factors is the electrical polarity of the workpiece being treated, which determines the nature of the vapour-gas shell (VGS) forming around it, which acts as a heat source and carrier of active particles. When a negative potential is applied (cathodic treatment), intense electrical discharges resembling a glow are observed, which at high current loads can transition to an arc mode. This significantly complicates the control of the product temperature, increases the risk of overheating and leads to surface destruction with an increase in micro-roughness. Regardless of polarity, when treated in aqueous electrolyte solutions containing VGS [6], a significant amount of water vapour is fixed. However, in the case of cathodic treatment, the level of material oxidation is significantly lower, which is probably due to the presence of hydrogen and the low content of free oxygen in the environment. The characteristic oxide layer during anodic EPT increases corrosion resistance but inhibits the diffusion of interstitial elements.

Additional interest in this technology is due to the possibility of activating diffusion processes through surface modification. For example, preliminary activation by means of ultra-deep penetration of SiC and Al particles allows for a significant intensification of boride layer formation and an increase in their thickness by 20-50% [7] due to the activation of volume diffusion and the formation of defect zones in the steel structure. Thus, under the harsh operating conditions of mining and metallurgical equipment, electrolytic-plasma boriding is considered a technologically and economically efficient alternate to traditional methods.

The aim of this work is to study changes in the structure, phase composition, microhardness and tribological parameters of 40Kh steel during diffusion electrolytic-plasma boriding (DEPB).

Materials and methods

Materials and methods In this research work, the objects of study are samples of 40Kh steel (AISI 5140) measuring 25×25×20 mm, which were pre-grinded with P2000 sandpaper before the experiments.

The experimental studies were conducted at the Surface Engineering and Tribology Research Centre of the S. Amanzholov East Kazakhstan University.

The experimental studies were conducted at the Research Centre for Surface Engineering and Tribology at S. Amanzholov East Kazakhstan University (Ust-Kamenogorsk, Kazakhstan). The microstructure of the samples was revealed by chemical etching using a 4% solution of nitric acid (HNO_3) in ethyl alcohol. The microstructure and energy-dispersive analysis of the samples were examined using a scanning electron microscope SEM3200 (China). The phase composition was determined using an X'PertPro X-ray diffractometer (Philips, Netherlands). Data processing and quantitative analysis were performed using PowderCell 2.4 software. To determine the depth hardness of the samples, a Vickers microhardness tester (Metolab 502, Russia) equipped with a diamond indenter and a load sensor up to 100 g was used.

The tribological characteristics of the boronised steel samples were evaluated by measuring the coefficient of friction and wear of the rubbing surfaces using the 'ball-on-disk' method on a TRB³ Anton Paar tribometer.

The diffusion electrolytic plasma boriding unit includes a direct current rectifier (Fig. 1a) that provides a direct current with a maximum power of 360 V and 100 A [9]. The electrodes consist of a cathode (workpiece) and an anode in the form of a round plate with holes for uniform distribution of the electrolyte. The electrolyte used is an aqueous solution containing calcined ash (Na_2CO_3) as an electrically conductive medium and saturating chemicals in specific proportions, which is considered optimal for the formation of stable plasma [10].

During electrolytic plasma treatment in the 'metal-electrolyte' system, a complex set of physical and chemical processes is implemented, initiated by the application of high electrical potential.

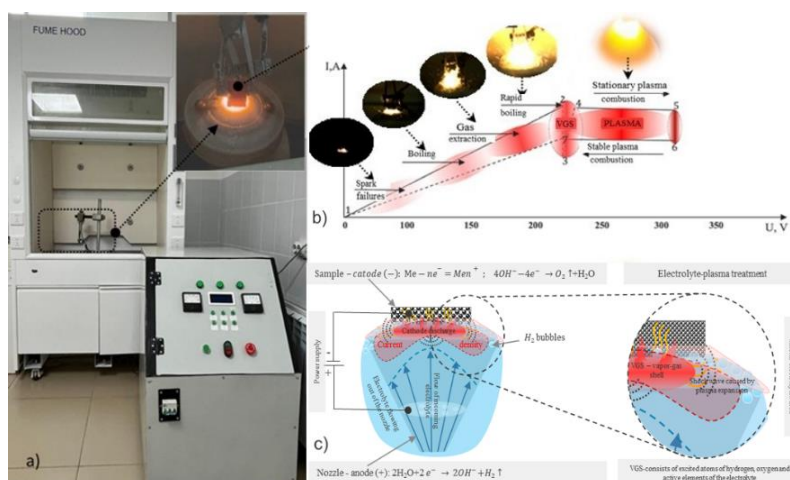


Figure 1 – General view of the installation and schematic diagrams of electrical discharge processes during electrolyte-plasma treatment (EPT):

a) general view of the DEPTB installation b) vapor-gas shell (VGS) c) diagram of electrical discharge

When heated gradually, the liquid first turns into a gas. If you keep heating the gas, ionisation happens: electrons separate from atoms, forming a mix of positively charged ions and free electrons. This ionised environment can conduct electricity. The process of gas transition to a plasma state is accompanied by a brief glow known as a glow discharge, during which the temperature can reach 2000°C. These high temperatures are effectively used to modify the surface of metallic materials using an external source of influence. The combination of electrolytic and plasma processes allows for the implementation of a surface treatment method known as electrolytic-plasma modification. The modification process can be divided into several phases. In Figure 1c, at voltages of 150-200 V, electrolysis occurs, accompanied by the release of gases (H_2 , O_2) (Fig. 1b) and the formation of gas bubbles. With a further increase in voltage above 200 V, a vapor-gas shell (VGS) forms at the interface, representing a dynamically unstable region of excited hydrogen and oxygen atoms and active electrolyte components. As the voltage increases to 250-300 V, stable plasma formation begins, accompanied by micro-discharges, sharp local heating of the sample surface (up to 1000 °C) and high temperature gradients (up to 500 °C/s). This leads to intensified mass transfer, increased diffusion and activation coefficients, and the formation of surface layers with modified structure and properties.

Thus, Figure 1d the mechanism of electrical discharge phenomena in EPT includes the following sequential stages: electrolysis → formation of VGS → discharge plasma, which are closely related to the technological parameters of processing (voltage, current, pulse duration) and determine the intensity of thermal and electrical impact on the metal.

To identify the phases formed in the coatings after high-temperature oxidation tests, X-ray diffraction (XRD) analysis was performed using an X'PertPRO diffractometer with Cu-K α radiation ($\lambda = 1.54 \text{ \AA}$) at 40 kV and 30 mA. Diffractograms were collected in the 2θ range of 20° to 90° with a step size of 0.02° and a counting time of 0.5 seconds per step. The data were analyzed using the HighScore software (Table 1).

Thermo-Calc software (version 2025a, Thermo-Calc Software, Stockholm, Sweden) was used to model the phase diagrams of boron-containing steels, which graphically display the phase transformations in iron-boron alloys depending on their composition and temperature.

Table 1 – DEPB modes for 40X steel

Sample	Voltage, V	Current, A	Temperature, °C	Time, min
40Kh	180	10	950	20
Electrochemical polishing	10	4,3	54	10
Electrolyte: 10% sodium carbonate + 20% sodium tetraborate + 70% water				

Electrochemical polishing was used to remove oxide layers from the surface of 40Kh steel samples before subsequent studies. Polishing was carried out in a cell with a stainless steel cathode at a constant voltage of 10-12 V and a current density of 1.4-1.6 A/cm² for 10 minutes at an electrolyte temperature of 50-60 °C.

Results and discussion

It is known [11-13] that alloying elements in most cases dissolve in austenite, forming solid solution substitutions. Alloyed steels require higher heating temperatures and longer holding times to obtain a homogeneous austenite in which the carbides of the alloying elements are dissolved. The low tendency for austenite grain growth is a technological advantage of most alloyed steels. All alloying elements reduce the tendency of austenite grains to grow, except for manganese and boron. Elements that do not form carbides (silicon, cobalt, copper, nickel) have little effect on grain growth. Figure 2 shows the Fe-B phase diagram (Figure 2a) [5] and the calculated model of M+B phase formation at different temperatures obtained using the ThermoCalc program (Figure 2b).

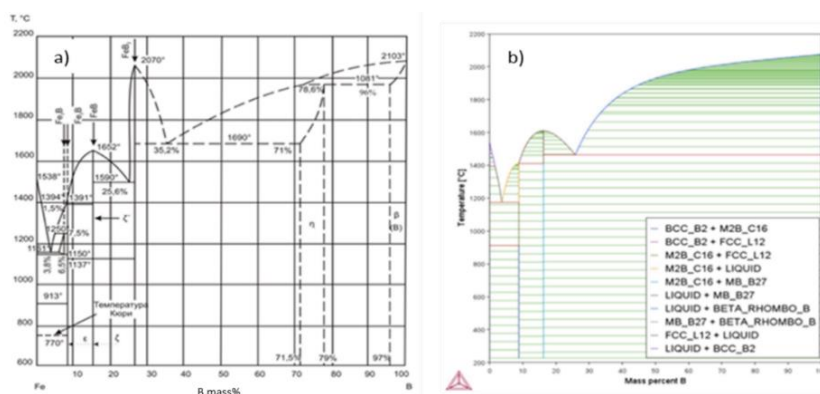


Figure 2 – Phase diagram of Fe-B as a function of processing temperature
a) the Fe-B phase diagram b) the Fe-B phase diagram obtained using the ThermoCalc program

Carbide-forming elements (chromium, molybdenum, tungsten, vanadium, titanium) greatly refine the grain. According to previous studies [13], it was established that the borided layer of low- and medium-alloy structural steel consists of borides FeB – η -phase (16.23% B) – surface layer, modification zone; Fe₂B – ϵ phase (8.83% B) – intermediate layer of the transition zone, adjacent to the core, i.e., in general, there is an alternation of structures: $\epsilon + \eta$; $\alpha + \epsilon$ phases.

Figure 3 shows fragments of the microstructure of the surface of a 40Kh steel sample treated with DEPB after electrochemical treatment to remove oxides formed during diffusion saturation. Numerous roughnesses were found on the surface, which is typical for electrolytic plasma treatment.

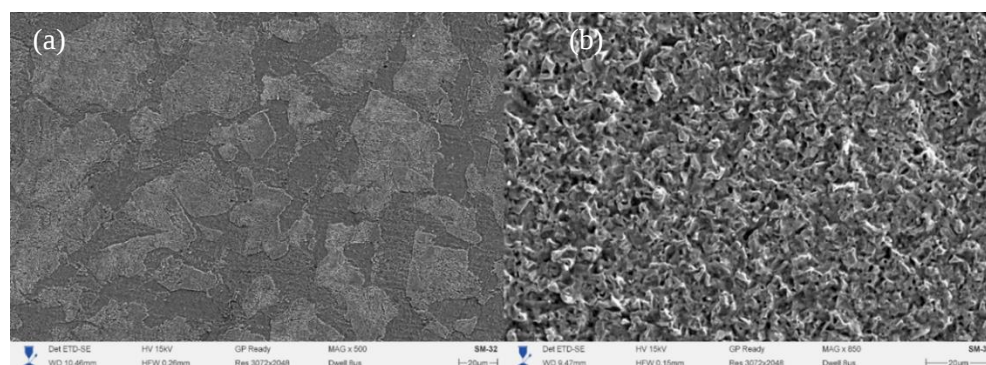


Figure 3 – Microstructure of the 40Kh steel before (initial) (a) and after DEPB treatment (b)

Figure 4 shows a SEM image of the diffusion layer of 40Kh steel borided by the plasma-electrolytic method. A quantitative elemental analysis was performed to determine the elemental composition. EDX analysis of the modified surface revealed that spectrum 1 contains 7.53% wt. and spectrum 3 contains 8.78% wt., while spectrum 2 contains 16.56 wt.% of boron mass concentrations (Table 2). According to the literature [14], the composition of typical FeB phases contains 16.23% wt. boron, and the Fe₂B phase should contain 8.83 wt.% boron, which confirms the obtained result (Table 2).

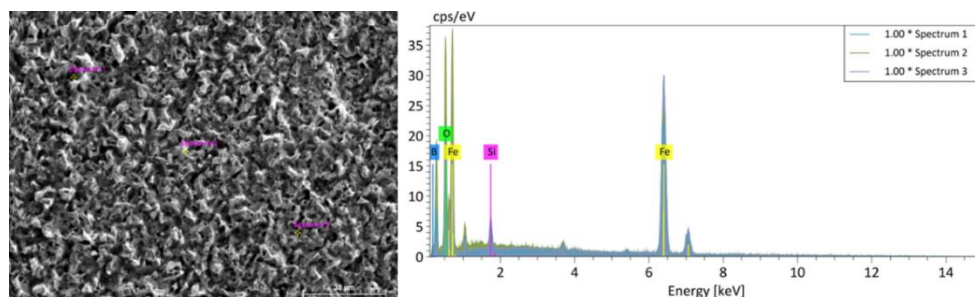


Figure 4 – Surface morphology and EDS analysis of 40X steel after DEPB

Table 2 – Chemical composition of 40Kh steel samples after DEPB steel (wt. %)

Spectrum	B	O	Si	Fe
Spectrum 1	7.53	14.91	5.59	71.97
Spectrum 2	16.56	18.78	1.29	63.37
Spectrum 3	8.78	11.09	1.37	78.76
Mean	10.96	14.93	2.75	71.37

The structure of the diffusion-borided surface layer of 40Kh steel consists of columnar crystals of iron borides FeB and Fe₂B. Figure 5 shows the cross-sectional microstructure of 40Kh steel after diffusion boriding in an electrolyte plasma. As can be seen from Figure 5 a, the modified surface layer has a zonal character: Layer I is a surface borided layer consisting of borides FeB (η -phase) and Fe₂B (ϵ -phase), the presence of which is confirmed by point elemental analysis (Fig 5b, c).

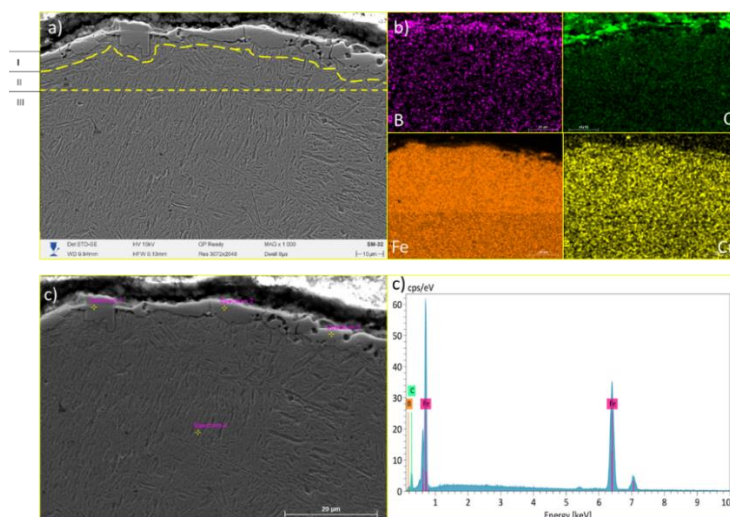


Figure 5 – Image of the cross-sectional microstructure of 40Kh steel after boriding, observed from the surface into the depth of the sample

a) SEM image of cross section of steel surface after DEPB: 1-Borided layer, 2– transitional intermediate layer, 3– thermal influence zone b,c,d) EDS analysis of 40X steel after DEPB

Layer II – transitional intermediate layer, dark component of the diffusion layer, formed from borides of alloying elements (Fe,Cr)₂B; Layer III – thermal influence zone. According to reference data [15], the resulting layer has high hardness, which provides increased abrasive wear resistance at high temperatures (up to 900°C).

Figure 6 shows the XRD pattern of 40Kh steel after plasma-electrolytic boriding [15] in an electrolyte containing an aqueous solution of 10% (Na_2CO_3) and 20% ($\text{Na}_2\text{B}_4\text{O}_7 \cdot 10\text{H}_2\text{O}$) at a temperature of 900 °C for 20 minutes. The results of X-ray phase analysis (XRD) are shown in Figure 6. For the initial sample of 40X steel, only peaks corresponding to α -Fe (reflections (110), (200), (211)) are observed. X-ray structural analysis confirmed that after diffusion boriding, additional reflections appear on the diffractogram of the studied steel, identified as iron borides – Fe_2B and FeB , while the intensities of the α -Fe peaks decreased significantly. The appearance of $\text{Fe}_2\text{B}/\text{FeB}$ indicates the formation of a boride layer on the surface due to boron diffusion and reaction with iron [15]. The partial overlap of the α -Fe(110) reflection with the boride reflections and the moderate broadening of the peaks indicate the small size of the boride layer crystallites and the occurrence of micro stresses.

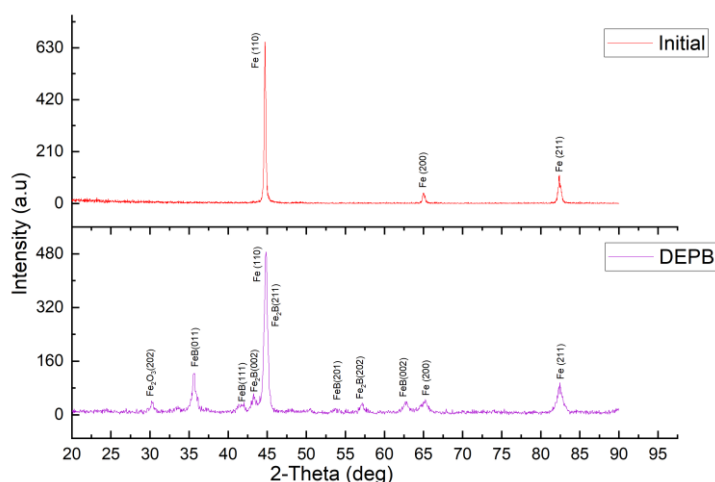


Figure 6 – XRD pattern of steel 40Kh steel after diffusion boriding

Unfortunately, the results of X-ray structural analysis did not reveal the presence of residual austenite phases during plasma-electrolytic boriding.

Only insignificant reflections corresponding to γ -phase particles were detected, which only indicates the presence of these phases in the material under study.

Friction tests showed that diffusion electrolyte-plasma boriding has a significant effect on the nature of changes in the friction coefficient of 40Kh steel (Figure 7) [16]. For the initial sample, a sharp increase from ~0.15 to ~0.86 is observed over the first 15 m of the path, which is associated with rapid wear and transition to a steady state of adhesive friction. For the borided sample, the increase in the friction coefficient is more gradual, reaching ~0.85 only at 47 m of the path.

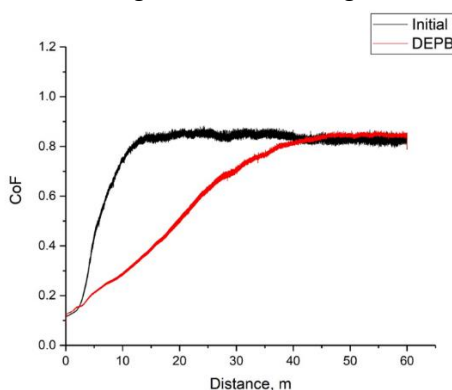


Figure 7 – Friction coefficient of 40Kh steel measured on a tribometer before and after DEPB

The long running-in stage and reduced friction coefficient values at the initial stage indicate the presence of a wear-resistant boride layer, which reduces the intensity of adhesive interactions and slows down surface destruction. The established friction coefficient value for both samples is close (~0.8), but the rate of reaching it is significantly lower for the boron-coated material, which indicates increased wear resistance and stability of the protective layer [17].

Conclusion

The studies conducted showed that diffusion-electrolyte-plasma boriding (DEPB) of 40Kh steel leads to the formation of a hard boride layer on the surface, consisting of FeB and Fe₂B phases, which is confirmed by the results of XRD phase analysis (Table 3).

Table 3 – Correlation table of the results of the study of 40Kh steel before and after DEPB

Sample	Phase	Microhardnes, HV	Coefficient of friction	Wear intensity, mm ³ /N·m
40Kh initial	Fe	236	0,764	2,276 E ⁻⁴
DEPB+polising	FeB Fe ₂ B	1250	0,611	6,492E ⁻⁵

As a result of boriding, the microhardness of the surface increased from 236 HV for the initial sample to 1250 HV, which is more than 5 times higher than the initial value.

Friction tests showed a decrease in the friction coefficient from 0.764 for the initial steel to 0.611 after DEPB, which indicates an improvement in the anti-friction properties of the surface. The wear rate decreased from 2.276×10^{-4} mm³/(N·m) to 6.492×10^{-5} mm³/(N·m), which indicates a significant increase in wear resistance.

Thus, the use of DEPB provides a comprehensive improvement in the operational properties of 40Kh steel due to the formation of a wear-resistant boride coating with high hardness and a reduced friction coefficient. The results obtained confirm the promise of this surface modification method for increasing the durability of parts operating under conditions of intense wear.

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ДИФфуЗИЯЛЫҚ ЭЛЕКТРОЛИТТІК-ПЛАЗМАЛЫҚ ӨНДЕУДІҢ 40Х БОЛАТТЫҢ ТРИБОЛОГИЯЛЫҚ СИПАТТАМАЛАРЫНА ӘСЕРІН ЗЕРТТЕУ

Бұл мақалада диффузиялық электролиттік-плазмалық борлаудың 40Х болатының құрылымы мен трибологиялық сипаттамаларына әсері қарастырылады. Рентгендік дифракция және сканерлеуші микроскопия әдістері арқылы құрылымдық-фазалық құрам зерттелді, ал болаттың механикалық және трибологиялық қасиеттері қаттылық өлшегіш пен трибометр арқылы бағаланды. 40Х болатының бастапқы күйі мен диффузиялық электролиттік-плазмалық өңдеуден кейін, оксидтерді электрохимиялық жылтырату арқылы жойғаннан кейінгі күйі салыстырылды. Диффузиялық электролиттік-плазмалық өңдеуден кейін рентгендік дифрактограммаларда FeV және Fe₂V боридтік фазалары анықталды, бұл диффузия процесінің жүргенін дәлелдейді. Сондай-ақ микроқаттылықтың 5 есеге артқаны және тозу интенсивтілігінің бастапқы күйге байланысты 3 есеге дейін төмендегені анықталды. Борлау процесінен кейінгі құрылым морфологиясы үш аймақтан тұратын зоналылыққа ие екені анықталды: борланған қабат, диффузиялық аймақ және термиялық әсер ету аймағы. Бұл зерттеу нәтижелері диффузиялық электролиттік-плазмалық өңдеудің машина жасау мен тау-кен өндірісі саласындағы бөлшектерді өңдеуде болашағы зор екенін көрсетеді.

Түйін сөздер: диффузиялық электролиттік-плазмалық өңдеу, борид, электролит, бура, микроқаттылық.

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ИССЛЕДОВАНИЕ ВЛИЯНИЯ ДИФфуЗИОННОЙ ЭЛЕКТРОЛИТНО-ПЛАЗМЕННОЙ ОБРАБОТКИ НА ТРИБОЛОГИЧЕСКИЕ ХАРАКТЕРИСТИКИ СТАЛИ 40Х

В этой статье рассмотрены влияния диффузионного электролитно-плазменного борирования на структуру и трибологические характеристики стали 40Х. Методами рентгеновской дифракции и сканирующей микроскопии были исследованы структурно-фазовый состав, а механические и трибологические свойства стали были оценены твердомером и

трибометром. Были сравнены характеристики стали 40Х в исходном состоянии и после диффузионной электролитно-плазменной обработки с последующим удалением оксидов электрохимическим полированием. После диффузионной электролитно-плазменной обработки на рентгеновских дифрактограммах были обнаружены боридные фазы FeB и Fe₂B, которые подтверждают процесс диффузии. Так же было определено повышение микротвердости в 5 раз и снижение интенсивности изнашивания до 3 раз в зависимости от исходного состояния. Установлено, что морфология структуры после борирования имеет зональность, состоящая из 3-зон: борированный слой, диффузионная зона и зона термического влияния. Данные исследования свидетельствуют о перспективности использования диффузионной электролитно-плазменной обработки в машиностроении и обработке деталей горно-рудной промышленности.

Ключевые слова: диффузионная электролитно-плазменная обработка, борид, электролит, бура, микротвердость.

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