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APPLICATION OF ELECTROLYTE BASED ON SODIUM CARBONATE FOR ELECTROLYTE-PLASMA SURFACE HARDENING OF STEEL 45

Abstract: *This article evaluates the electrolyte-plasma hardening (EPH) of steel 45 using an electrolyte based on sodium carbonate (Na_2CO_3). Theoretical foundations of the electrolysis process of a sodium carbonate aqueous solution were studied during the course of the work. The mechanical and tribological properties of 45 steel samples after EPH were studied. It was found that with an electrolyte composition of 20% Na_2CO_3 and 80% distilled water, a hardened zone with a thickness of up to 5 mm is formed after EPH. The increase in microhardness reached up to 690 HV, which corresponds to a 3–3.5 times increase compared to the initial state. The results of tribological tests showed that the friction coefficient of steel 45 decreased after EPH, indicating a significant improvement in tribological characteristics compared to the initial value before hardening. Electrochemical tests on the corrosion resistance of steel 45 were also carried out. After EPH, the corrosion rate significantly decreased for steel 45 samples, indicating its highest corrosion resistance.*

Key words: *Electrolyte-plasma hardening, steel 45, structure, wear resistance, microhardness.*

Introduction

Electrolyte-plasma hardening (EPH) is a promising thermal treatment method that involves applying a plasma discharge to the surface of steel in an electrolyte environment at high temperatures. During EPH, an electrolysis process also occurs, which is an ion exchange reaction between the anode and the cathode. Various surface modifications can be achieved depending on parameters such as electrode size, applied voltage, and electrolyte chemical composition. Due to its simplicity and environmental friendliness, EPH represents a promising technology for surface modification of materials. Several studies have focused on the impact of EPH technology on steel properties, highlighting its ability to improve surface properties by enhancing hardness, wear resistance, and corrosion resistance [1-4].

During EPH, plasma is generated under the influence of an electric arc, creating a high-temperature environment where physicochemical processes involving ionized gas particles occur. In electrolyte-plasma quenching, the treated material is intensely heated within the vapor-gas shell formed around it, followed by power shutdown and washing with the electrolyte, leading to structural changes in the steel to a certain depth. Additionally, during electrolyte-plasma treatment, besides the thermal impact of plasma on the steel surface, a diffusion saturation process can occur, where ions and electrons penetrate the metal's surface layer, causing changes in its structure and chemical composition. This advantage of EPH allows for the diffusion saturation of steel with atoms of carbon, nitrogen, and boron, and alloying with metals. Due to differences in chemical composition, the depth of plasma penetration may vary for different steels, ultimately affecting the hardening efficiency and uniformity [5-6].

The electrolysis process is influenced by the composition of the electrolyte, the surface area of the cathode and anode, and the cooling rate during electrolyte-plasma quenching. Previous studies have shown that among various electrolyte compositions containing an aqueous solution of sodium carbonate (Na_2CO_3) at concentrations of 15%, 20%, and 25%, the 20% electrolyte composition provides the best mechanical and tribological properties for steel 45 during EPH.

Sodium carbonate, when interacting with water, dissociates in two stages, leading to the formation of sodium bicarbonate and sodium hydroxide:

$$2Na^+ + CO_3^{2-} + HOH \rightleftarrows Na^+ + HCO^{3-} + Na^+ + OH^-$$

$$Na^+ + HCO_3^- + HOH \rightleftarrows H_2CO_3 + Na^+ + OH^- \qquad (1)$$

This change in ion concentration affects the specific conductivity of the solution, which varies according to a parabolic relationship with a maximum at its peak. This dependence is expressed by the following equation

$$\sigma = \alpha CZF(u_+ + u_-) \qquad (2)$$

where α is the degree of dissociation, C is the molar concentration, Z is the valence, F is the Faraday constant, and u_+, u_- – are the ionic mobilities of cations and anions, respectively. Equation (2) applies to singly charged ions. The decrease in conductivity at low concentrations is explained by the minimal influence of ions on each other, manifesting through electrophoretic and relaxation effects [7-8]. As ion concentration increases, their interaction becomes more pronounced. However, after reaching a certain concentration of Na_2CO_3, the specific conductivity decreases despite the increase in the total number of ions. This occurs because, as ions move in opposite directions, the frictional effect becomes predominant, restricting their free movement. According to equation (2), the specific conductivity of the electrolyte reaches its maximum at a concentration of 30%, with values of σ = 36,61 Ом-1×см-1for the first stage and σ = 53,01 Ом-1×см-1 for the second stage. As seen from equation (1), more ions are produced during the first stage of hydrolysis, leading to greater mutual influence among them. Upon reaching the second stage of hydrolysis, the free bicarbonate ion binds with a hydrogen ion. As a result, the total number of free ions in the solution decreases, allowing for freer movement of the remaining ions, which in turn increases the specific conductivity. However, in practice, hydrolysis in the second stage occurs either weakly or not at all. In Figure 1, the theoretical values of specific conductivity at a temperature of $t = 18\,^oC$ are shown in black. The graph demonstrates that as the maximum point approaches, the rate of conductivity increase diminishes, and further increases in concentration do not lead to significant growth in conductivity. This behavior of electrolyte conductivity with 20% Na_2CO_3 content makes this composition optimal for the rapid ignition of plasma within the vapor-gas shell during EPH, which, in turn, ensures efficient and quick heating of the sample.

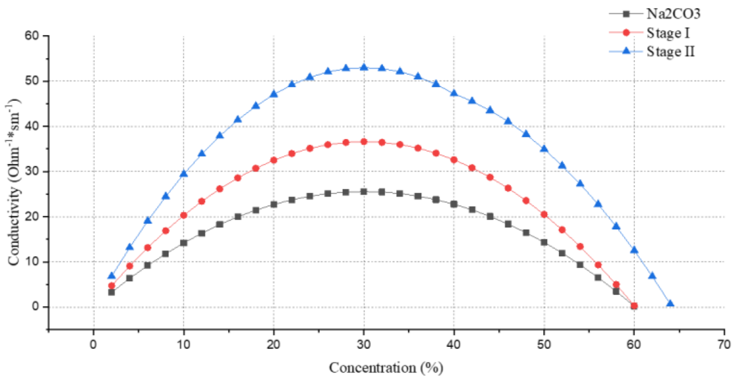

Figure 1 – Dependence of the specific conductivity of Na_2CO_3 electrolyte on concentration at two stages of hydrolysis

In the near-electrode zone, the heat balance is maintained by the release of heat flow and its absorption by the metallic electrode and the liquid electrolyte. The sources of the heat flow include the chemical reactions occurring in the system and the heating of the vapor-gas shell according to the Joule-Lenz law:

$$Q = \sum \Delta H_i + \Delta UI, \qquad (3)$$

where $\sum \Delta H_i$ represents the heat flow released during chemical reactions, and ΔU is the voltage drop across the vapor-gas shell. It is also noted that the contribution of the heat flow from the Ohmic component is more significant compared to other components of the heat source. This is due to the low conductivity of the vapor-gas shell, which results in a large voltage drop ΔU in this section of the electrical circuit: DC power source – anode – electrolyte – vapor-gas shell – cathode. The power generated according to the Joule-Lenz law can vary within the range of 0.1-1 MW/cm². The basic setup of the EPH installation and its simplified electrical circuit are shown in the figure [9-12].

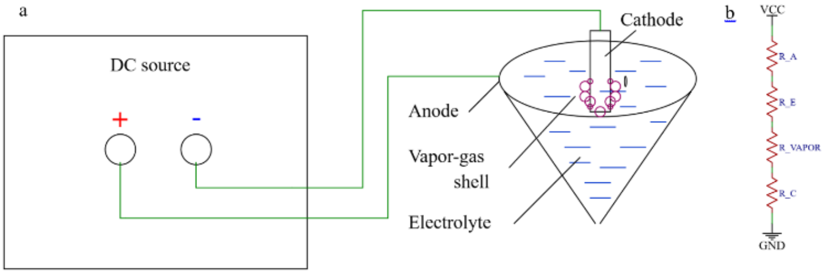

Figure 2 – Schematic diagram of the EPH installation – a; and its simplified electrical circuit – b: R_A – anode, R_E – electrolyte, R_VAPOR – vapor–gaseous shell, R_C – cathode

The Na_2CO_3-based electrolyte also possesses sufficient critical cooling speed, which, combined with the high heat flow, enables thermal hardening of steel. This technology allows for the formation of a surface layer with enhanced mechanical and corrosion properties, while the steel's core retains its original ductility and impact strength. This makes the technology preferable for various applications, including the hardening of working parts of agricultural machinery. Due to the high energy flow, EPH can accelerate the treatment of steel products, unlike induction hardening, which requires much longer processing times, leading to increased costs for the entire technological process. Additionally, during induction hardening, the workpiece must be held very close to the inductor, which becomes more challenging when using high-frequency currents. Another widely used surface hardening technology, flame hardening, can cause deformation of the part during treatment. Although both flame and induction hardening can produce a uniform fine-grained microstructure with good mechanical properties, the aforementioned drawbacks limit their use in some practical applications [13-15].

EPH using an aqueous solution based on Na_2CO_3 is capable of forming not only martensitic phases on the steel surface but also phases of retained austenite and the formation of special carbides, as confirmed by the results of transmission electron microscopy analysis conducted by

Popova N. and others. The EPH process was carried out at a voltage of 320V and a current of 40A, with a heating duration of 4 seconds. The resulting surface layer provides excellent mechanical properties, such as increased hardness and wear resistance [16-17].

In the work by Kenesbekov A. et al., the results of electrolyte-plasma quenching of 40KhN steel using an aqueous Na_2CO_3 solution as an electrolyte at a voltage of 200-350 V are presented. The researchers note that after 3 seconds of heating, the microhardness doubled, reaching 440 HV, and the wear resistance increased by 70% compared to the initial sample. Additionally, after EPH, a fine-grained martensitic microstructure with carbide inclusions and retained pearlite formed on the steel surface, which positively affects the improvement of the mechanical properties of the surface [18-19].

In another study [20], the application of a 20% aqueous Na_2CO_3 solution for surface EPH of 20GTL steel samples is discussed. The EPH process used direct current with a voltage of 360V and a current of 60A in a flowing electrolyte, with rapid heating in 2-3 seconds. After the power was turned off, the samples were cooled in circulating electrolyte. The results of measurements of the mechanical and tribological properties of the steels also confirm the effectiveness of using Na_2CO_3 in EPH: microhardness doubled, and the wear volume decreased by 25% [21].

Thus, it can be said that Na_2CO_3 is an optimal component for the electrolyte in EPH, providing sufficient electrical conductivity and critical cooling rate. Additionally, research shows that EPH is the most economical and efficient method, characterized by low energy consumption, simplicity of technological equipment, large sizes of the hardened zone, high process productivity, less environmental harm, and the ability to harden parts of large mass and complex profile. Consequently, this solves the problem of rapid wear and reduced efficiency of machine components and mechanisms, which is especially important under conditions of increased operational loads [22].

The aim of this work is to evaluate the effectiveness of a Na_2CO_3-based electrolyte in providing optimal conductivity and critical cooling rate during the electrolyte-plasma quenching of steel 45, which are crucial for improving its mechanical and tribological properties. The selection of this material as the subject of the study is justified by its wide application in mechanical engineering and its high mechanical characteristics, such as strength and wear resistance, which can be significantly enhanced through EPH. The results of this study will contribute to the refinement of EPH parameters and provide valuable data on the potential of the technology to extend the service life of heavily loaded machine parts.

Materials and Methods

Experimental studies and tests were conducted at the Research and Innovation Center for Instrumental and Technological Development of VKU named after S. Amanzholov (Ust-Kamenogorsk, Kazakhstan) and the Engineering Center "Hardening Technologies and Coatings" at Shakarim University in Semey, Kazakhstan.

Steel 45 was selected as the research object. The samples were prepared by grinding and polishing before the experiment. EPH of the samples was carried out using an electrolyte-plasma processing unit [23] under three different regimes, as presented in Table 1. For all samples, an electrolyte consisting of a 20% aqueous solution of Na_2CO_3 was used.

Table 1 – Parameters of electrolytic-plasma hardening of samples made of steel 45

Sample	Anode Area, cm²	Voltage during Heating, V	Voltage after Heating, V	Current during Heating, A
№ 1	200	320	0	70
№ 2	500	320	10	100
№ 3	500	320	0	100

Metallographic analysis was conducted using an HL-102AW metallographic microscope equipped with a 3.0 MP digital camera and analysis software. For metallographic microanalysis, the samples were etched with a 3% alcohol solution of nitric acid after polishing with chromium oxide paste. Microhardness measurements were performed using a Vickers HV-1 DT hardness tester with a load of 1 N on the indenter and a dwell time of 10 seconds, in accordance with GOST 9450-76.

Morphology of the treated samples was studied using a JSM-6390LV scanning electron microscope from JEOL (Japan) with an energy-dispersive microanalysis attachment, INCA Energy, from OXFORD Instruments.

Tribological tests were carried out using an Anton Paar TRB3 tribometer following the "ball-on-disk" scheme. Test parameters included a wear radius of 3 mm, a friction path of 60 mm, a sample rotation speed of 2 cm/s, and a load of 6 N. A 100Cr6 material ball with a diameter of 6 mm was used as the counter body.

Electrochemical analysis was employed to study the corrosion resistance of the samples during the experiment. The evaluation of corrosion resistance helps determine which type of steel is more resistant to corrosion under specific operating conditions. The electrochemical corrosion study was conducted using a CS300M apparatus, which combines the functions of a potentiostat and galvanostat. An electrochemical cell was used to measure the corrosion rate depending on the potential at the working electrode. For this, the potentiostat was connected to the cell via three different cables: a silver chloride electrode (RE) as the reference electrode, a platinum electrode (CE) as the auxiliary electrode, and the sample (WE) as the working electrode. This method allows the study of the influence of potential on the corrosion rate and the evaluation of the degree of protection of materials against corrosion under various conditions. A 0.5 M NaCl solution was used for corrosion resistance testing. This solution was divided into separate containers, which were connected using a salt bridge. This setup creates uniform exposure conditions for the samples while maintaining a consistent chemical environment for corrosion testing. The CS Studio6 software was used to determine the potential values, current density, and corrosion rate, as well as to calculate Tafel slopes (ba, bc). The test results are presented as graphs on a logarithmic current density scale.

Phase composition analysis of the samples was performed using an X'PertPro X-ray diffractometer from Philips (Netherlands) with CuKα radiation. The Bragg-Brentano configuration was used for measurements. Data processing and quantitative analysis were carried out using X'Pert HighScore Plus software (version 3.0).

Results and Discussion

The study of the structural-phase states of the hardened surface layers of steel 45 before and after EPH revealed significant changes. The microstructure of steel 45 samples before and after EPH is presented in Figure 3.

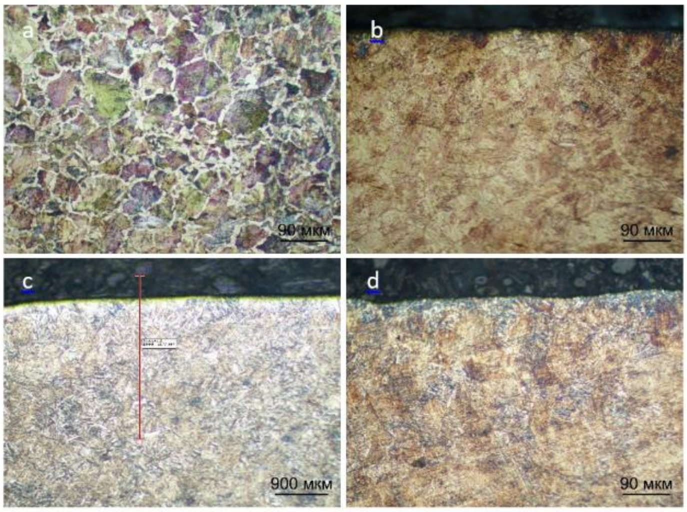

Figure 3 – Optical micrographs of steel 45: a – before; b, c, d – after EPH

The optical metallographic analysis revealed that in the initial state (Fig. 3a), the structure of steel 45 is characterized as ferritic-pearlitic, with a predominance of pearlite grains over ferrite. The EPH process led to the formation of a fine-dispersed martensitic structure (Fig. 3b, c, d) and significantly increased the hardness and wear resistance of the surface. The formation of a needle-like martensitic microstructure is also confirmed by the SEM images shown in Figure 4 [23, 24].

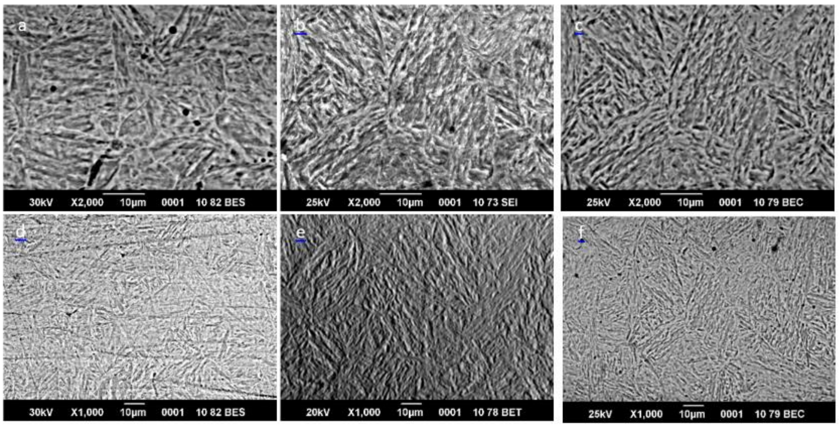

Figure 4 – SEM micrographs of steel 45 samples at magnifications of ×2000 and ×1000:
a, d – № 1; b, e – № 2; c, f – № 3

The results of microhardness measurements of the surface layer of steel 45 before and after EPH are shown in Figure 5. The initial microhardness of steel 45 is 209 HV. The greatest increase in microhardness is observed in sample No. 3, where it reaches 690 HV, corresponding to a 3-3.5 times increase compared to the initial state. It was also observed that the thickness of the hardened layer was 5 mm.

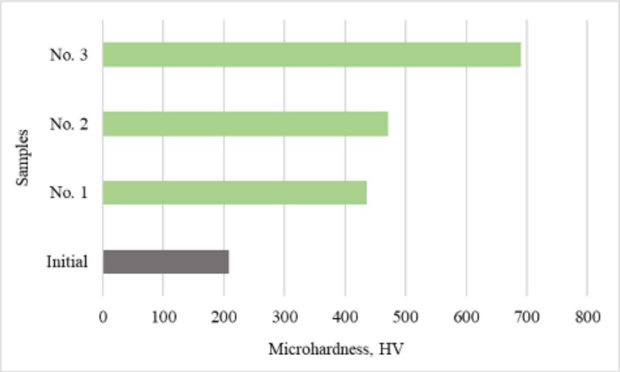

Figure 5 – Microhardness results of steel 45 samples before and after EPH

Thus, EPH significantly increased the surface microhardness of steel 45, particularly for sample No. 3, demonstrating the effectiveness of this method in enhancing the wear resistance of the material.

Additionally, experiments were conducted to evaluate the changes in the tribological characteristics of medium-carbon steel 45 before and after EPH. Tribological tests were performed using the «ball-on-disk» method. Figure 6 presents the friction coefficient versus sliding distance graphs for the steel 45 samples after EPH [25]. Sample No. 3 shows the lowest friction coefficient of 0.454, indicating the greatest improvement in tribological characteristics among all samples, with a 1.5 times enhancement compared to the initial values before hardening.

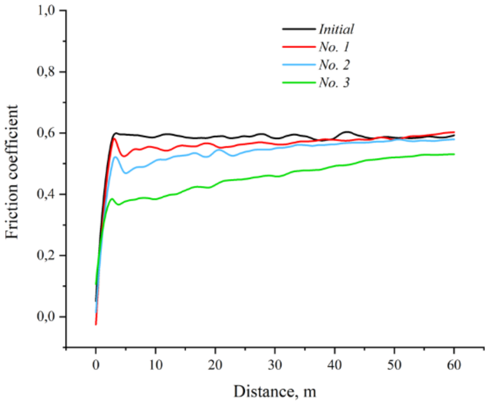

Figure 6 – Friction coefficient vs. sliding distance graphs for steel 45 samples before and after EPH

The X-ray diffraction (XRD) pattern shown in Figure 7 displays peaks corresponding to the crystalline phases of sample No. 3. The main peaks on the graph are identified as α´-Fe, indicating the presence of martensite in the sample. The peaks at 2θ values of approximately 44°, 65°, and 82° correspond to the primary diffraction lines for ferrite with crystallographic plane indices (110), (200), and (211), respectively.

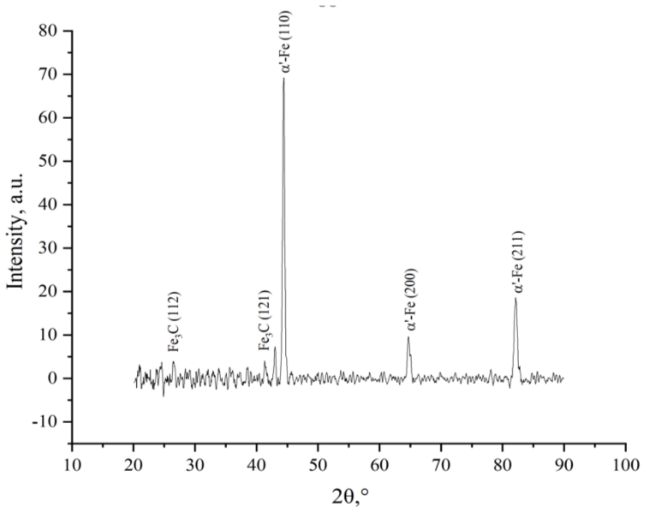

Figure 7 – X-ray diffraction (XRD) pattern of sample № 3

Additionally, peaks corresponding to the Fe_3C carbide phase were detected. Overall, the X-ray diffraction analysis confirms the presence of phases typical for quenched steels, which explains the increased hardness and wear resistance of the samples after EPH.

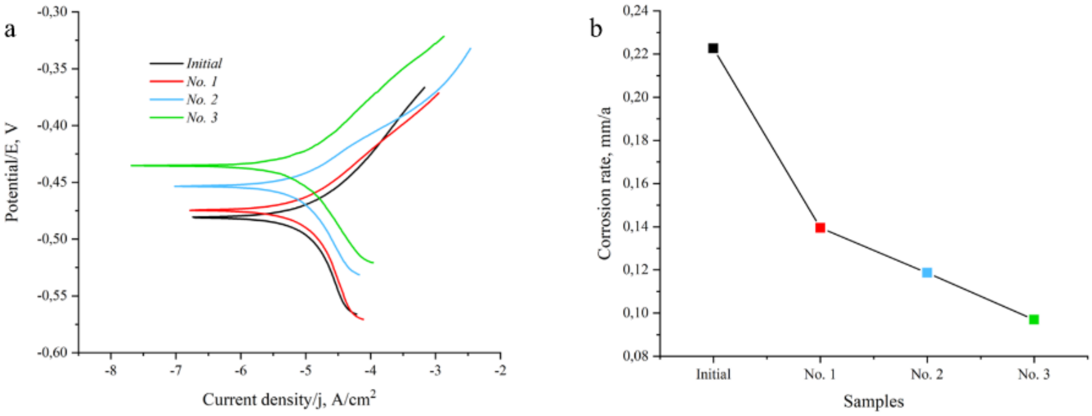

Figure 8 – Potentiodynamic polarization curves for electrochemical corrosion resistance test – a, and corrosion rate graph of samples before and after EPH – b

The presented polarization curve (Figure 7a) shows the relationship between current density and potential for samples before and after EPH (Initial, №1, № 2, № 3). Samples № 1, № 2, and № 3 exhibit a shift in corrosion potential towards more positive values and a decrease in corrosion current density compared to the initial sample, indicating an improvement in their corrosion resistance. Sample № 3 shows the lowest corrosion current density (8.2602×10^{-3} mA/cm^2), which suggests the best corrosion resistance among all the samples studied. Therefore, electrolyte-plasma surface hardening significantly enhanced the corrosion resistance of samples № 1, № 2, and № 3, with sample № 3 showing the highest effectiveness. Additionally, Figure 7b presents the corrosion rate values for the four samples: Initial, № 1, № 2, and № 3. The initial sample has the highest corrosion rate (0.22255 mm/a), indicating the lowest corrosion resistance among all the samples studied. After EPH, the corrosion rate significantly decreases for samples № 1, № 2, and № 3. The best result is observed in sample № 3, with a corrosion rate of 0.096902 mm/a, indicating the highest corrosion resistance.

Conclusion

The conducted study demonstrated that EPH of steel 45 using a Na_2CO_3-based electrolyte significantly enhances its mechanical, tribological, and corrosion properties. The hardened zone up to 5 mm thick formed after EPH resulted in an increase in microhardness to 690 HV, which corresponds to a 3-3,5 times increase compared to the initial state. The reduction in the friction coefficient after EPH indicates a significant improvement in the tribological characteristics of steel 45. Additionally, the decrease in the corrosion rate after EPH points to the high corrosion resistance of the treated samples. Therefore, EPH can be considered an effective method for improving the performance characteristics of steel 45.

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БОЛАТТЫҢ ЭЛЕКТРОЛИТ-ПЛАЗМАЛЫҚ БЕТІН ҚАТАЙТУ ҮШІН НАТРИЙ КАРБОНАТЫ НЕГІЗІНДЕГІ ЭЛЕКТРОЛИТТІ ҚОЛДАНУ 45

Бұл мақалада натрий карбонаты негізіндегі электролит көмегімен 45 болаттың электролит-плазмалық қатаюы бағаланады. Жұмыс барысында натрий карбонатының сулы ерітіндісін электролиздеу процесінің теориялық негіздері зерттелді. Электролиттік плазманың қатаюынан кейінгі 45 болат сынамасының механикалық және трибологиялық қасиеттері зерттелді. Электролиттік құрамы 20% натрий карбонаты мен 80 тазартылған судың электролиттік-плазмалық қатаюынан кейін қалыңдығы 5 мм-ге дейін шыңдалған аймақ түзілетіні анықталды. Микроқаттылықтың жоғарылауы 690 ВВ ДЕЙІН болды, бұл бастапқы күймен салыстырғанда 3-3, 5 есе өсуге сәйкес келеді. Трибологиялық сынақтардың нәтижелері ЭПҚ-дан кейін 45 болаттың үйкеліс коэффициентінің төмендегенін көрсетті, бұл сөндіруге дейінгі бастапқы мәнмен салыстырғанда трибологиялық сипаттамалардың айтарлықтай жақсарғанын көрсетеді. Сондай-ақ 45 болаттың коррозияға төзімділігіне электрохимиялық сынақтар жүргізілді. EPU - дан кейін болат 45 үлгілері үшін коррозия жылдамдығы айтарлықтай төмендеді, бұл оның коррозияға ең жоғары төзімділігін көрсетеді.

Түйін сөздер: *электролиттік плазманың қатаюы, болат 45, құрылымы, тозуға төзімділігі, микроқаттылығы.*

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ПРИМЕНЕНИЕ ЭЛЕКТРОЛИТА НА ОСНОВЕ КАРБОНАТА НАТРИЯ ДЛЯ УПРОЧНЕНИЯ ЭЛЕКТРОЛИТНО-ПЛАЗМЕННОЙ ПОВЕРХНОСТИ СТАЛИ 45

В данной статье оценивается электролитно-плазменное затвердевание стали 45 с использованием электролита на основе карбоната натрия. В ходе работы были исследованы теоретические основы процесса электролиза водного раствора карбоната натрия. Исследованы механические и трибологические свойства 45 проб стали после отверждения электролитической плазмы. Установлено, что после электролитически-плазменного отверждения 20% карбоната натрия и 80 дистиллированной воды образуется закаленная зона толщиной до 5 мм. Увеличение микротвердости составило до 690 ВВ, что соответствует увеличению в 3-3, 5 раза по сравнению с исходным состоянием. Результаты трибологических испытаний показали снижение коэффициента трения стали 45 после ЭПУ, что свидетельствует о значительном улучшении трибологических характеристик по сравнению с исходным значением до затухания. Также были проведены электрохимические испытания коррозионной стойкости стали 45. После EPU скорость коррозии значительно снизилась для моделей стали 45, что свидетельствует о ее максимальной коррозионной стойкости.

Ключевые слова: *электролитическое плазменное упрочнение, сталь 45, структура, износостойкость, микротвердость.*

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