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Investigation of the Influence of Technological Regimes of Thermocyclic Electrolyte-Plasma Treatment On The Structural-Phase State and Tribocorrosion Properties of 12Kh1MF Steel

This paper presents the results of a detailed study on the influence of thermocyclic electrolyte-plasma treatment on the structural-phase state, microhardness, and tribocorrosion properties of heat-resistant steel 12Kh1MF. The treatment was carried out using a 10 % Na_2CO_3 aqueous solution and a voltage of 300/150 V. It resulted in the formation of a zonal microstructure with a martensitic surface layer up to 600 μm thick. The structure was divided into zones: hardened, thermal influence, and base material. Microhardness increased by 1.5 times due to martensitic transformation. X-ray analysis confirmed the formation of α′Fe and Fe_3C phases. Tribological tests showed a 10 % reduction in the friction coefficient and a 1.5–2-fold increase in wear resistance. Corrosion tests in a salt fog chamber revealed enhanced protective properties, with mass loss reduced and corrosion resistance improved by 10–30 %, depending on the treatment mode. Based on a comprehensive analysis, the most effective thermocyclic treatment mode was identified, ensuring an optimal balance of mechanical strength and corrosion resistance. The findings highlight the high potential of thermocyclic electrolyte-plasma treatment as a promising method for enhancing the surface properties of steel components operating in aggressive environments.

Keywords: electrolytic-plasma treatment, heat-resistant structural steel, thermocyclic electrolytic-plasma treatment, microhardness, low-alloy steel, microstructure, surface strengthening, corrosion testing

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Introduction

Structural steels are materials widely used in power engineering and other industries. They have a number of characteristics that make them suitable for use in high temperature and pressure environments. In thermal power plants, they are used to manufacture steam pipes, turbines and other types of parts. However, with prolonged service life, degradation of the structure occurs, which leads to unstrengthening of the steel [1]. In such cases for steel hardening the traditional heat treatment is carried out, which is carried out with volumetric treatment of the entire working surface of the part, but as it is known in working mechanisms the core of the part must remain plastic for proper operation of the entire unit [2]. Therefore, scientists and engineers develop various methods of hardening, including not only classical heat treatment, but also additional methods that can modify only the near-surface area of parts without changing its plastic characteristics. Technologies based on electro-discharge machining processes, such as electrolyte-plasma technologies for material modification, can be considered as an example of such machining methods [3]. Combining these methods can increase the strength, wear resistance and heat resistance of structural steels, providing longer service life in extreme conditions.

Modern research in the field of electrolyte-plasma processing is actively developing, covering both theoretical and practical aspects of electrodischarge phenomena, contributing to the creation of highly efficient and cost-effective methods of materials processing such as electrical discharge machining (EDM) [4, 5], electrospark machining (ESM) [6], electrolyte-plasma hardening (EPH) [7], and electrolyte-plasma anodic treatment (EPAT) [7, 5], electrospark machining (ESM) [6], electrolyte-plasma hardening (EPH) [7], electrolyte-plasma anodic treatment (EPAO) [8], electrolyte-plasma chemical heat treatment (EPChT) [9] expanding their application in various industrial sectors, including power and mining engineering.

The study by Garba E., Abdul-Rani A.M., Yunus N.A. [10] and co-authors demonstrates the wide possibilities of electrical discharge machining (EDM), which is used in such industries as mold and die manufacturing, as well as in mechanical engineering, nuclear, aerospace and biomedical industries. According to the authors' conclusions, the main advantage of this technology is the high accuracy of manufacturing complex parts. However, EDM has a number of significant disadvantages, including low material removal rate, poor surface quality, long processing time, high cost, as well as being limited to processing only electrically conductive materials. These factors limit its application in production.

In the monograph by Korzhyk V., Tyurin Yu. and Kolisnichenko O. [11] the results of theoretical and experimental studies of dynamic processes of modification of metal surfaces by means of electric current regulated by means of an electrode with liquid electrolyte were presented. Based on the conducted studies, integrated hardening technologies were developed and specialized equipment was created, allowing for oxidation, alloying and structural transformation of the surface without affecting the entire volume of the product. The use of these technologies significantly improves the physical and mechanical characteristics of materials, increasing their strength, wear resistance and resistance to fatigue loads under friction and wear conditions.

The studies by Berladir K. and her co-authors consider the process of nitrocarburizing with cyclic heating in comparison with isothermal nitrocarburizing [12]. The authors found that the use of thermocyclic nitrocarburizing in comparison with isothermal nitrocarburizing allows for an increase in resistance to cavitation destruction by 0.6–1.5 times. In addition, the use of chemical-thermocyclic treatment helps to reduce processing time, reveal the grain structure and improve the mechanical properties of structural steel.

One of the promising technologies in this area is thermocycling electrolytic-plasma treatment (TEPT). This method combines thermal, electrical and plasma effects on the material in an electrolytic environment, where active elements contribute to its modification due to the flow of electric current through the environment to the surface of the material. This approach allows to significantly increase the hardness of the surface layers. The TEPT process includes cyclic alternation of heating and cooling, which causes significant structural changes in the material and improves its mechanical properties, facilitating wide application in production. Thus, according to the analysis, it was established that technologies based on the processes of electric discharge phenomena are applicable as an alternative to traditional heat treatment in order to improve the performance characteristics of a wide class of steels, however, the definition of technological regimes of cathodic electrolytic-plasma treatment with thermocycling has not been studied in detail. Therefore, the aim of this work is to study the influence of technological regimes of thermocyclic electrolytic-plasma treatment on the structural-phase state and tribocorrosion properties of 12Kh1MF structural steel.

Materials and methods of research

In this work, 12Kh1MF steel was used as the material for TEPT. This steel is widely used in the power industry, in particular, it is used to manufacture pipelines, boilers and other elements operating in aggressive environments. The chemical composition of 12Kh1MF steel according to GOST 20072-74 is given in Table.

T a b l e

Chemical composition of steel grade 12Kh1MF, %

C	Si	Mn	Cr	Mo	V	P	S
0.1–0.15	0.17–0.37	0.4–0.7	0.9–1.2	0.25–0.35	0.15–0.03	>0.035	>0.03

Thermocyclic hardening of 12Kh1MF steel samples was performed using an electrolytic plasma treatment unit at the Surface Engineering and Tribology Research Center of the Sarsen Amanzholov East Kazakhstan University. The EPO unit schematically consists of an electrolytic cell and a power source (Figure 1 *a, b*).

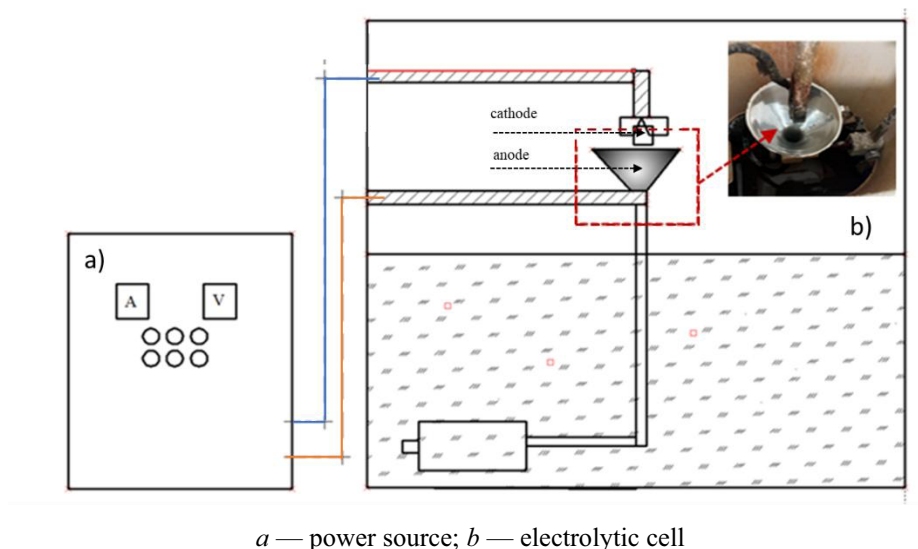*a* — power source; *b* — electrolytic cell

Figure 1. Schematic representation of the unit for thermocycling electrolytic-plasma treatment

The essence of the process of thermocycling hardening by the method of electrolytic-plasma treatment is as follows. At low voltage (about 150 V) in the electrolytic cell with an aqueous solution of electrolyte, a standard electrochemical process occurs. With an increase in voltage to 300 V, intensive gas evolution begins on the electrodes, which leads to the formation of a vapor-gas shell (VGS) near the electrode surface.

The electric field strength in the VGS reaches 10^4–10^5 V/cm. At a temperature of about 100 °C, such a field initiates the ionization of vapors, as well as the emission of ions and electrons necessary to maintain a stable electric discharge. As a result, electrolyte plasma is formed. In this process, an aqueous solution of sodium carbonate (Na_2CO_3) acts as a medium for heating and cooling.

For thermocyclic electrolytic-plasma heating of 12Kh1MF steel, an aqueous solution of sodium carbonate (Na_2CO_3) with a concentration of 10 % and 15 % (wt. %) was used. Our previous studies have established [13, 14] that at higher concentrations of Na_2CO_3, the electrical conductivity of the electrolyte increases, which leads to intense heating and high current density. A 10 % aqueous solution of Na_2CO_3 has a low current density compared to 15 % Na_2CO_3, which ensures smoother heating without the risk of melting the steel surface, and also ensures effective control of the heating process for various technological tasks. Therefore, in further experiments, we will use an electrolyte composition with 10 % sodium carbonate and 90 % distilled water.

During thermocycling (Table 2), when an electric potential of 300 V is applied, the sample surface rapidly heats up. When periodically switching between high electric potential (300 V) and low (150 V), a cyclic change in the heating rate is observed. This regime allows controlling the intensity of the thermal effect, which helps to increase the duration of heating and form a thick heated layer on the surface of the material. By alternating supply at a voltage of 300 V and a current of 30 A for 5 seconds, with a decrease to 150 V and a current of 8 A for 5 seconds, the electrolyte temperature increases to 37 °C. This allows controlling the heating process, providing different temperature profiles and intensity of thermal effect on the samples.

T a b l e 2

Results of corrosion tests of samples before and after TEPT

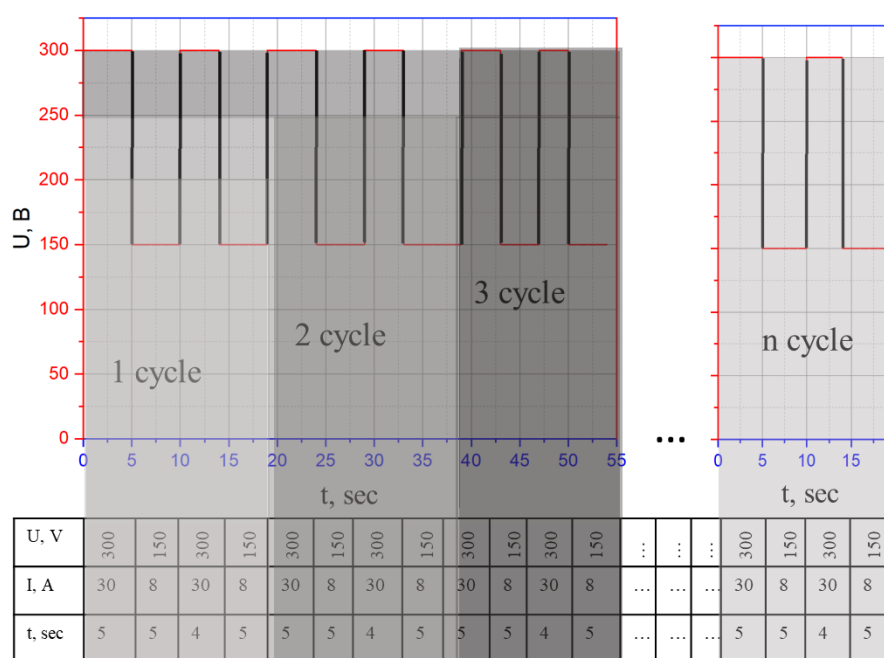| Sample	Mass beforetesting, g	Mass aftertesting, g	Loss of mass, g	Loss of mass per unit area Δm, kg/m^2
Initial	9.5391	9.5299	0.0092	0.0191
Cycle No.1	11.2293	11.2198	0.0095	0.0129
Cycle No.2	8.4946	8.4883	0.0063	0.0109
Cycle No.3	7.0022	6.9941	0.0081	0.0152

Figure 2. Parameters of the thermocycling electrolytic plasma treatment regime

The maximum steel surface temperature reaches 900 °C in the third cycle, with an increase in the electrolyte temperature to 39 °C. In cycles № 1 and № 3, the steel surface temperature reaches 800 °C and 850 °C, respectively, with the electrolyte temperature increasing to 36 °C and 37 °C, respectively.

The samples for processing were ground and polished on the Metapol-2000P unit. The microstructure of the samples was revealed by chemical etching using a 4 % solution of nitric acid (HNO_3) in ethyl alcohol. The microstructure of the original and processed steels was studied on a TESCAN MIRA scanning electron microscope with a magnification of ×60 and ×1000.

X-ray phase analysis of the samples to determine the phase composition before and after TEPT was carried out on an Expert Pro unit. Surface analysis was carried out from 10° to 90° with a delay of 0.02 s, in 2θ mode.

To determine the hardness of the cross-section of the samples, a METOLAB 502 microhardness tester was used, equipped with a tetrahedral Vickers diamond pyramid with a square base and an angle of α = 136° between the opposite faces at the apex in strict compliance with the requirements of GOST 9450-76, imposed on the Vickers method. The diamond indenter under a load of $F = 1$ N was pressed perpendicularly and maintained under load for 10 s.

Tribological tests were carried out on a TRB3 tribometer. At a load of 6 N over a distance of 60 m using the ball-on-disk method and a speed of 2 cm/s. The radius of the trace was 3 mm ($D = 6$ mm). A ball with a diameter of 6 mm made of 100Cr6 (analogous to ShKh15) was used as a counterbody.

Corrosion resistance tests were carried out in a HUD-E808 salt fog chamber according to GOST 34388-2023. The neutral salt medium was 5 % sodium chloride (NaCl). The study was conducted at a temperature of 35 °C for 8 hours. Although the actual working conditions of 12Kh1MF steel in steam pipelines typically involve temperatures of 400–550 °C in a water vapor medium, such laboratory conditions are commonly used for accelerated corrosion testing. These parameters allow the simulation of long-term surface degradation processes under controlled and reproducible conditions, providing a comparative evaluation of corrosion resistance before and after surface treatment.

Quantitative assessment of corrosion was carried out according to GOST 9.908-85. The mass loss per unit surface area Δm, g/m^2, was calculated using formula (1):

$$\Delta m = \frac{m_0 - m_1}{S}, \tag{1}$$

where m_0 is the mass of the sample before testing, g; m_1 is the mass of the sample after testing and removal of corrosion products, g; S is the surface area of the sample, m^2.

Results and discussion

Figure 3 shows the SEM results of the microstructure of the cross section of 12Kh1MF steel after treatment in an electrolyte containing an aqueous solution of 10 % Na_2CO_3 in three different cycles (No. 1, No. 2, No. 3), which allows us to draw conclusions about the influence of thermal conditions on the formation of the structure and tribomechanical properties of materials. In the initial state, 12Kh1MF steel has a ferrite-pearlite structure. After TEPT in different cycles, the structure of 12Kh1MF steel mainly consists of the martensite phase, which is associated with rapid cooling, which leads to an increase in microhardness and an improvement in tribocorrosion properties (Table 2, 3).

After TEPT under different conditions, zonal structures are formed in 12Kh1MF steel, which differ in microstructure and hardness, and are distinguished as the zone of thermocycling quenching, the heat-affected zone and the original material (Fig. 3).
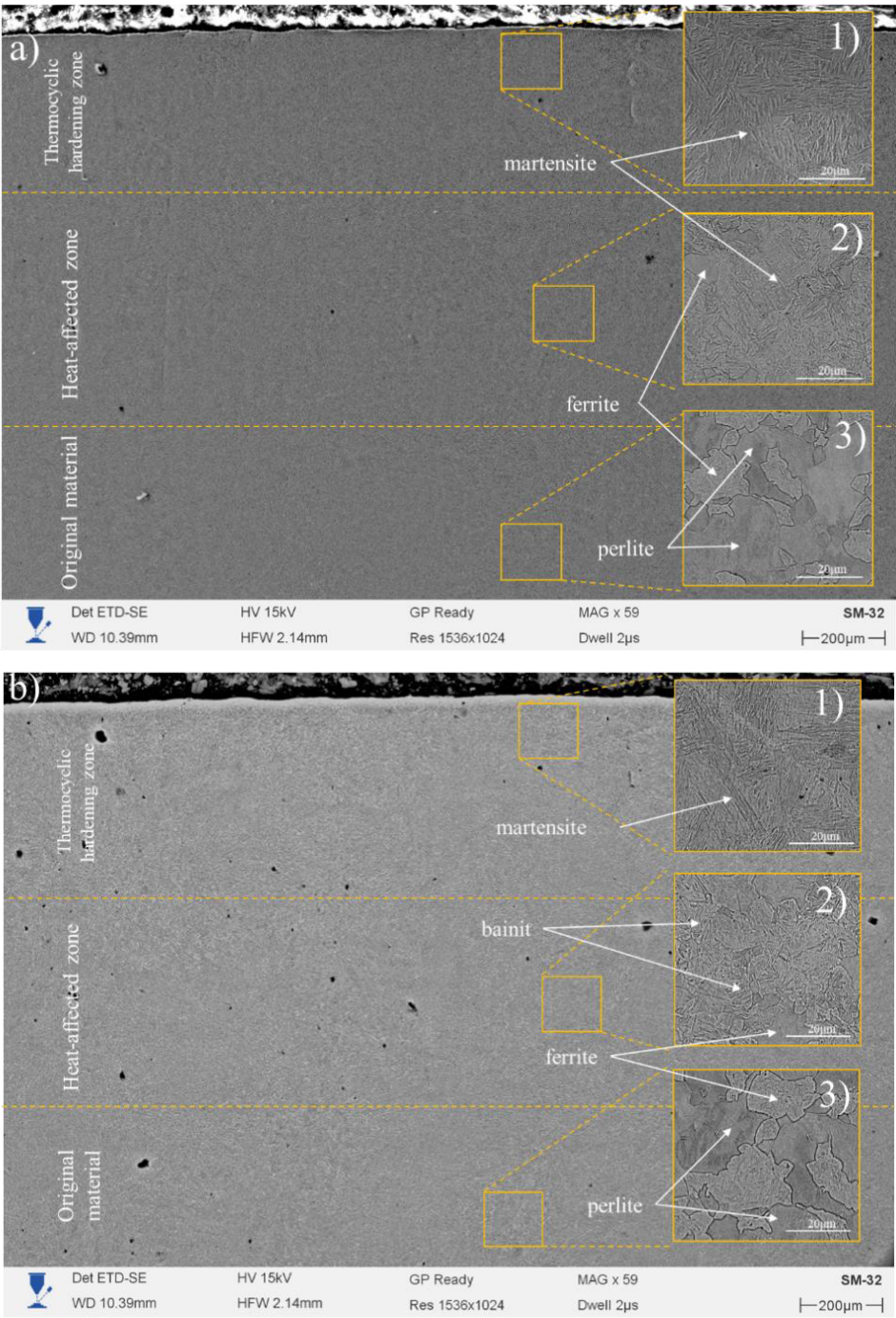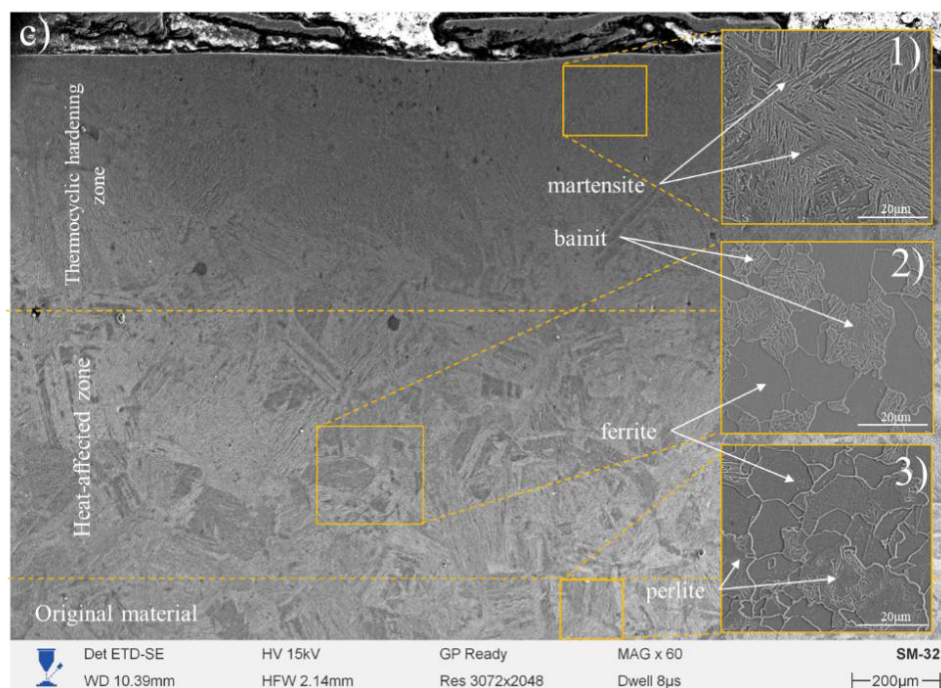
Figure 3. SEM image of the cross-section of 12Kh1MF steel after TEPT in cycles No. 1 (*a*), No. 2 (*b*) and No. 3 (*c*):
1) thermocyclic hardening zone; 2) heat-affected zone; 3) original material

As shown in Figure 3*a*, the microstructure of 12Kh1MF steel obtained after TEPT in cycle No. 1 is obvious that the zone of thermocycling quenching is characterized by the presence of a martensite microstructure with a thickness of ~400 μm (Fig. 3, *a*, 1), while the heat-affected zone demonstrates the transition from martensite to the original state (ferrite-pearlite) (Fig. 3, *a*, 2, 3). In cycle No. 2, a zone with a thickness of ~500 μm is also visible (Fig. 3, *b*, 1). The formation of martensite is a consequence of rapid cooling. In the zone of thermocycling quenching, the martensite structure predominates, and in the heat-affected zone, bainite and ferrite (Fig. 3, *b*, 2, 3). In cycle No. 3, the martensite microstructure with a thickness of ~600 μm in the zone of thermocycling quenching transforms into bainite, thereby entering the heat-affected zone with a thickness of ~600 μm (Fig. 3, *c*, 1, 2, 3). Thus, the zone of thermocycling quenching under different cyclic conditions has a thickness of up to 600 μm with a martensitic structure that contributes to the improvement of the surface microhardness of the material.

Table 3 shows the results of measuring the surface microhardness of steel samples before and after TEPT. According to the results, it was found that after TEPT, the microhardness increased by ~1.5 times, which is associated with the formation of a martensitic structure.

T a b l e 3

Results of corrosion tests of samples before and after TEPT

| Sample | Phasecomposition | Microhardness, $HV_{0.1}$ | TRB[3] | | Corrosionrate, mm/year |
			μ	Wear intensity, $mm^3/N \times m$	
Initial	αFe	205±30	0.627	0.0004706	0.0024
Cycle No.1	$\alpha`Fe$, Fe_3C	345±30	0.617	0.0004391	0.0016
Cycle No.2	$\alpha`Fe$, Fe_3C	365±34	0.596	0.0003047	0.0013
Cycle No.3	$\alpha`Fe$, Fe_3C	373±31	0.613	0.0002672	0.0019

In order to identify changes in the structural and phase state, X-ray phase analysis of the surface of 12Kh1MF steel samples was carried out before and after TEPT under different conditions (Fig. 4). According to the presented results of X-ray phase analysis, it was found that in the initial state, the steel has a ferrite-pearlite structure (αFe phase) (Fig. 4), and after TEPT, the formation of a martensite phase ($\alpha`Fe$) with a strengthening cementite phase (Fe_3C) is observed in all samples, which is consistent with the results of SEM analysis.
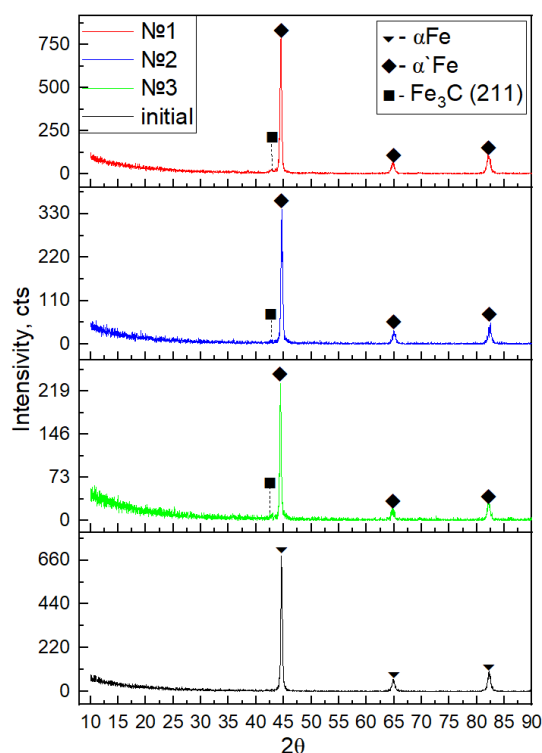
Figure 4. Results of X-ray phase analysis of steel before and after TEPT in different cycles

Figure 5 shows the results of tribological tests of steel samples before and after TEPT, according to which it was revealed that, compared to the initial state with a value of 0.627, the friction coefficient decreased by an average of 10 %, and the wear resistance of 12Kh1MF steels increased by ~1.5–2 times (Table 3).
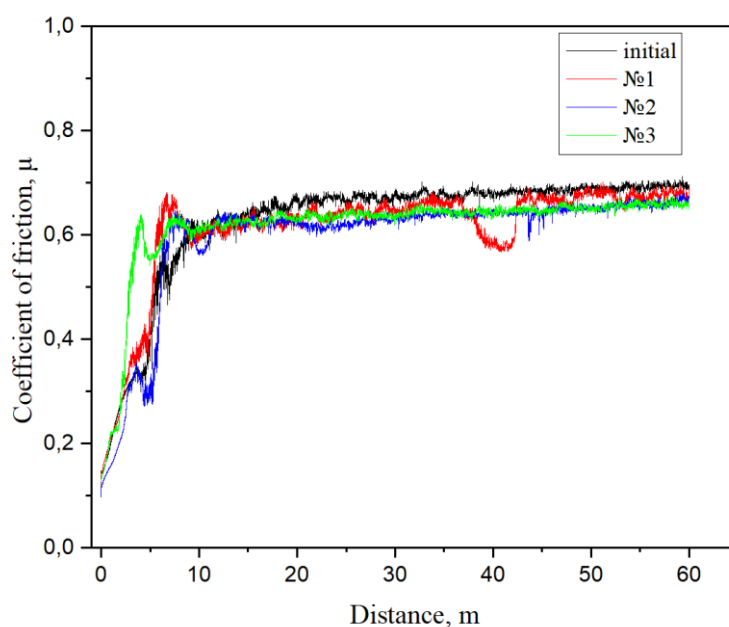

Figure 5. Tribology results before and after TEPT

The results of corrosion tests of steel before and after treatment are presented in Table 2. According to the analysis of the mass loss of samples using formula (1), it was found that after testing, the corrosion resistance improved by 10–30 % compared to the original sample. A reduction in mass loss is observed in cy-

cle № 2 when compared to cycles № 1 and № 3. It is evident that the more moderate regime of cycle № 2, in comparison to cycle № 3, serves to reduce the likelihood of residual stresses that have the potential to accelerate localised corrosion.

Thus, the results of the studies show that the concentration of the electrolyte and the selected heating cycles have a significant effect on the properties and structure of steels. The results of the studies and their analysis are correlated in Table 3.

It has been established that the electrolyte concentration (10 % Na_2CO_3) in combination with optimal heating cycles (e.g. No. 2 and No. 3) contributes to an increase in microhardness and improvement of tribocorrosion properties due to stable energy density and controlled heating during TEPT.

Conclusion

On the basis of the obtained results the following deductions and conclusions were made:

– the microstructure of the cross section of steel 12Kh1MF after TEPT has a zonal structure, it was found that in the electrolyte containing 10 % Na_2CO_3 + 90 % distilled water, is formed thermocyclic hardened layer with a thickness of up to 600 microns, which varies depending on the cycle, structurally consisting of the main phases of martensite (α`Fe) and cementite (Fe_3C), the formation of which contributes to an increase in microhardness in ~1.5 times.

– According to the results of tribo-corrosion tests of samples before and after TEPT it was revealed that wear resistance of the samples was increased by ~10 % on average, and corrosion resistance improved by 10– 30 % compared to the original sample.

On the basis of the obtained results the optimal technological regime of thermocyclic electrolyte-plasma treatment of steel 12Kh1MF was established: electrolyte composition aqueous solution of 10 % sodium carbonate at a temperature of 900 °C, 2-3 cycles of treatment at a voltage of 300 V – 150 V and a current of 30 A – 8 A for 5–60 seconds.

Acknowledgments

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Термоциклдік электролитті-плазмалық өңдеу технологиялық режимдерінің 12Х1МФ болатының құрылымдық-фазалық күйіне және трибокоррозиялық қасиеттеріне әсерін зерттеу

Жұмыста 12Х1МФ маркалы ыстыққа төзімді болаттың құрылымдық-фазалық күйіне, микроқаттылығына және трибокоррозиялық қасиеттеріне термоциклдік электролиттік-плазмалық өңдеудің әсері жан-жақты зерттелді. Өңдеу 300/150 В кернеуде натрий карбонатының (Na$_2$CO$_3$) 10 % сулы ерітіндісінде жүргізілді. Нәтижесінде қалыңдығы 600 мкм-ге дейін жететін мартенситтік құрылымды зоналық микроқұрылым қалыптасты. Микроқұрылым ұшы мынадай аймақтарға бөлінді: термиялық циклді қатайту, жылу әсер ететін аймақ және бастапқы материал. Мартенситтік құрылымның түзілуіне байланысты болаттың микроқаттылық қабілеті бастапқы материалмен салыстырғанда 1,5 есе өсті. Рентгендік фазалық талдау α'Fe және Fe$_3$C фазаларының түзілуін анықтады. Трибологиялық сынақтар үйкеліс коэффициентінің 10% төмендегенін және тозуға төзімділігінің 1,5-2 есе артқанын көрсетті. Тұзды тұман камерасындағы коррозиялық сынақтар материалдың қорғаныс қасиеттерінің жақсарғанын көрсетті: масса жоғалту азайды және өңдеу режиміне байланысты коррозияға төзімділік 10–30 %-ға артты. Жүргізілген талдау нәтижесінде әдістің 12Х1МФ конструкциялық болатының беріктік пен коррозияға төзімділіктің оңтайлы теңгерімін қамтамасыз ететін ең тиімді термоциклдік өңдеу режимі анықталды. Алынған нәтижелер термоциклдік электролиттік-плазмалық өңдеудің агрессивті ортада жұмыс істейтін болат бөлшектерінің беткі қасиеттерін жақсартуда тиімді әдіс екенін дәлелдейді.

Кілт сөздер: электролитті-плазмалық өңдеу, ыстыққа төзімді құрылымдық болат, термоциклдік электролитті-плазмалық өңдеу, микроқаттылық, төмен қоспаланған болат, микроқұрылым, беттік беріктендіру, коррозияға сынау

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Исследование влияния технологических режимов термоциклической электролитно-плазменной обработки на структурно-фазовое состояние и трибокоррозионные свойства стали 12Х1МФ

В данной работе всесторонне исследовано влияние термоциклической электролитно-плазменной обработки на структурно-фазовое состояние, микротвердость и трибокоррозионные свойства жаропрочной стали марки 12Х1МФ. Обработку проводили в 10 % водном растворе карбоната натрия (Na$_2$CO$_3$) при напряжении 300/150 В. В результате образовалась зональная мартенситная микроструктура толщиной до 600 мкм. Микроструктура была разделена на зоны наконечника: разделена на зону термоциклического упрочнения, зону термического воздействия и исходный материал. За счет образования мартенситной структуры микротвердость стали увеличилась в 1,5 раза по сравнению с исходным материалом. Рентгенофазный анализ выявил образование фаз α'Fe и Fe$_3$C. Трибологические испытания показали снижение коэффициента трения на 10 % и увеличение износостойкости в 1,5–2 раза. Коррозионные испытания в камере солевого тумана показали улучшение защитных свойств материала: уменьшение потери массы и повышение коррозионной стойкости на 10–30 % в зависимости от режима обработки. В результате проведенного анализа был определен наиболее эффективный режим термоциклической обработки конструкционной стали 12Х1МФ, обеспечивающий оптимальный баланс прочности и коррозионной стойкости. Полученные результаты доказывают, что термоциклическая электролитно-плазменная обработка является эффективным методом улучшения поверхностных свойств стальных деталей, работающих в агрессивных средах.

Ключевые слова: электролитно-плазменная обработка, жаропрочная конструкционная сталь, термо-циклическая электролитно-плазменная обработка, микротвёрдость, низколегированная сталь, микро-структура, упрочнение поверхности, коррозионные испытания

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