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Impact of Volume and Surface Heat Treatment on the Structure and Properties of Steel 30HGSA

The work presents the results of a comparative study of volumetric and surface heat treatment impact on the structural-phase states, hardness, and wear resistance of steel 30HGSA. Surface hardening was conducted by the electrolyte-plasma method. Bulk quenching of the samples was carried out by heating to a temperature of 900 °C, followed by cooling in water and oil, and some of the samples after quenching were annealed at a temperature of 510 °C. The structural-phase states of 30HGSA steel samples were studied by metallographic and X-ray structural analysis. There were carried out the microhardness measurements, tribological tests according to the ball-disk scheme, as well as was determined the resistance of the samples to abrasive wear. It was determined that after electrolytic-plasma hardening, fine-acicular martensite with a small content of cementite is formed on the basis of metallographic and X-ray structural analyzes, and coarse-acicular martensite is formed after volume quenching in water and oil. It was determined that the microhardness increased to 400-460 HV after volume quenching, and subsequent annealing leads to a decrease in hardness to 330-360 HV. It was revealed that the electrolyte-plasma surface hardening leads to an increase in microhardness up to 2 times due to the formation of fine-acicular martensite.

Keywords: hardening, annealing, electrolytic plasma treatment, volume quenching, structure, wear resistance, martensite.

Introduction

One of the most effective ways to improve the service characteristics of structural steels is the development of optimal heat-treatment modes. This facilitates to obtain products with certain specified characteristics that meet operational requirements, and, on the other hand, to predict changes in the properties of parts and structures during manufacture and operation [1-3]. The high level of physical and mechanical properties of structural steels, widely used in industry, is due to the martensitic structure [1]. They are subjected to bulk or surface hardening to obtain a martensitic structure in steels. An important place in increasing the durability of a wide class of machine parts is given to the quality of the metal — not of the entire section of the product, but to the structural state and physical and mechanical properties of the surface layer. It is the surface layer that determines the operational properties of parts — wear resistance, strength, material resistance to fatigue, contact endurance, corrosion resistance, etc. Surface treatment technologies with concentrated energy flows are widely used to increase the durability of parts, which have a number of common features that distinguish them favorably from other heat treatment methods. These technologies include the following methods of surface hardening: hardening by electric current induced in the surface layers of the part (HFC or induction hardening); flame hardening of an oxygen-acetylene or gas burner; plasma hardening; hardening by a laser beam; electric arc hardening; quenching in molten metals, electrolytes or salts [4-6]. Among them, plasma hardening has a number of advantages over the existing methods of surface thermal hardening in terms of its technical and economic indicators and the results of comparative analysis. The main advantage of plasma thermal hardening in comparison with laser is that the area of contact of the plasma arc with the material being processed is much larger than that of the laser beam; therefore, less time is spent on processing the surface of the die tooling with this method [3, 6]. One of the promising methods of plasma surface hardening is electrolytic-plasma hardening [7-10], which is one of the varieties of plasma hardening. Heating and cooling of the part is carried out in a water-based electrolyte during electrolytic-plasma hardening. The plasma layer is formed in the gap between the liquid (electrolyte) electrode and the

surface of the product when voltage is applied [10]. The result of a short stay of steel at quenching temperatures, as well as the occurrence of phase transformations in the temperature range above equilibrium, is an increase in the mechanical properties of the material in comparison with bulk quenching. However, the tribological properties of steels treated by electrolytic-plasma hardening have not been sufficiently studied, and there is little information on comparative studies of the wear resistance of steels treated with volumetric heat treatment and surface electrolytic-plasma hardening.

In connection with the above, the purpose of this work is to comparatively research the impact of volumetric and surface heat treatment on the structural-phase states and physical-mechanical properties of steel 30HGSA.

Experimental

Structural alloy steel 30HGSA was chosen as the object of research. Table 1 illustrates the chemical composition of the researched steel.

Table 1

Chemical composition of 30HGSA steel, %

Steel	C	Si	Mn	Ni	S	P	Cr	Cu
30HGSA	0.28-0.34	0.9-1.2	0.8-1.1	till 0.3	till 0.025	till 0.025	0.8-1.1	till 0.3

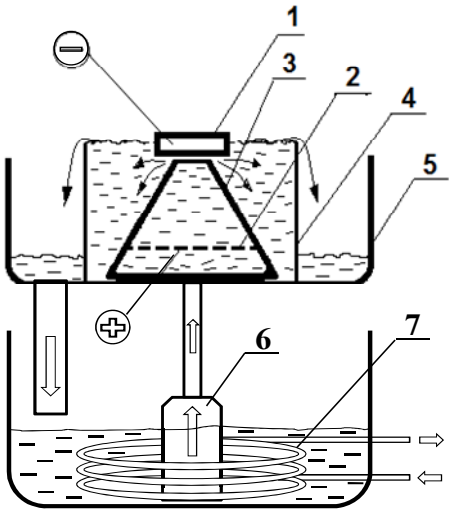
Samples of 30HGSA steel with the size of 15x15 mm were subjected to volumetric and surface heat treatment. The heat treatment modes are represented in Table 2. Heat treatment was carried out in an evacuated quartz tube in a laboratory tube furnace. Heat treatment of 30HGSA steel samples was carried out in the following modes: quenching from 900 °C, holding time at 900 °C was 0.5 h, cooling was conducted in water and oil, and some of the samples were subjected to annealing at 510 °C with cooling in water and oil. Surface hardening of 30HGSA steel samples was carried out by the electrolytic-plasma method on a setup consisting of a 30 kW direct current source, an electrolytic cell, a bath, a pump, a heat exchanger, and a stainless steel anode [11].

Table 2

Modes of processing 30HGSA steel samples

Sample designation	Volume quenching			Annealing			Surface hardening		
	Heating temperature, T, °C	Holding time t, sec	Cooling environment	Heating temperature, T, °C	Holding time t, sec	Cooling environment	Heating temperature, T, °C	Holding time t, sec	Cooling environment
Q0	-	-	-	-	-	-			
EPH	-	-	-	-	-	-	2 sec	-	water solution
Q1	900	30	water						
Q2	900	30	oil						
Q1A1	900	30	water	510	30	water			
Q2A1	900	30	oil	510	30	water			
Q1A2	900	30	water	510	30	oil			
Q2A2-	900	30	oil	510	30	oil			

The EPH process was carried out in the following mode: the applied voltage between the anode and the sample was 320 V, the current density was 25 A/cm^2, and the plasma exposure time was 2 sec. In this mode, the samples were heated to ~ 850-900 °C. Cooling was carried out in a flowing electrolyte after turning the voltage off. An aqueous solution containing 15% sodium carbonate was used as the electrolyte. Distilled water was used to prepare the electrolyte. The schematic view of the installation for plasma electrolytic processing is shown in Figure 1.



1 - processed sample (cathode), 2 - stainless steel anode with holes, 3 - cone-shaped baffle, 4 - working chamber - electrolyte bath, 5 - pan, 6 - pump, 7 - heat exchanger

Figure 1. Schematic view of the installation of plasma electrolytic treatment

The research of the phase composition of 30HGSA steel samples before and after volumetric and surface hardening was carried out on an X'PertPRO X-ray diffractometer in CuKα-radiation in a continuous recording mode in the range of angles from 20 to 85°. Metallographic analysis was performed in a bright field on an Altami MET 5C microscope at various magnifications. The microhardness of the samples was measured by the method of indentation of a diamond indenter on a PMT-3M device in accordance with GOST 9450-76, at a load of 100 g and holding under a load of 10 s. Tribological tests were carried out on a TRB^3 tribometer with dry friction according to the "ball-disk" scheme (ASTM G133-95 and ASTM G99) under the following conditions: wear radius – 3 mm, friction path – 60 m, sample rotation speed – 2 cm/s , a load of 6 N. Si_3N_4 ball with a diameter of 6 mm was used as a counterbody. Figures 2a-b represent a schematic of the experiment and a general view of the TRB^3 tribometer.

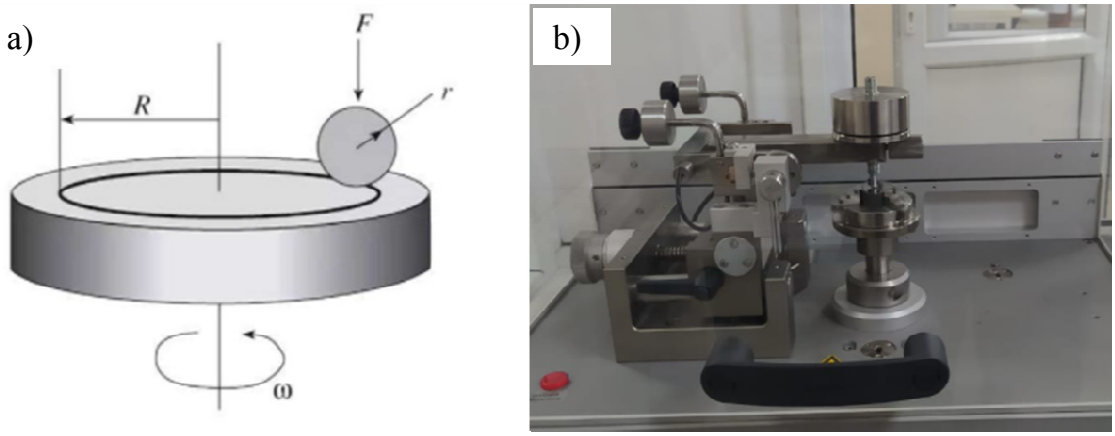


Figure 2. Experiment a schematic (a) and general view of TRB^3 tribometer (b)

Abrasive wear tests of the samples were carried out on an experimental setup for testing abrasive wear when rubbing against loose abrasive particles according to the "rotating roller – flat surface" scheme in accordance with GOST 23.208-79, which coincides with the American standard ASTM C 6568. The surfaces of the samples were ground and polished for testing abrasion on a rubber disc, they were also cleaned with acetone, and dried. A cylindrical rubber roller, pressed by a radial surface against a flat surface of a test specimen with a force of 44 N, rotated at a frequency of 1 s^{-1}. The general and schematic view of the device is shown in Figure 3a-b. The rate of entry of abrasive particles between the rubber wheel and the sample, that is, into the test zone, was 41-42 g/min. Electrocorundum with a grain size of 200-250 μm was used

as abrasive particles. The wear resistance of the treated test piece was evaluated by comparing its wear with that of a reference piece (non-treated piece). The wear was measured by the gravimetric method on an ADV-200 analytical balance with an accuracy of 0.0001 g. The samples were tested for 10 minutes, the total wear length was 96 m, and then, were blown with compressed air to remove the remaining sand particles on the samples before weighing. The wear resistance of the test material was assessed by the loss of the mass of the samples during the test in accordance with GOST 23.20879.

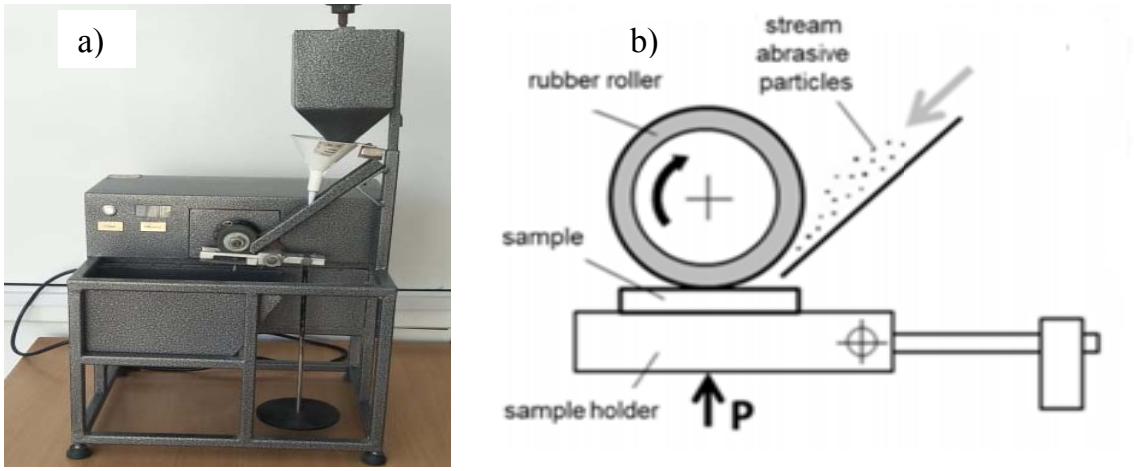


Figure 3. Experiment a schematic (a) and general view (b) of the device for testing materials for abrasive wear

Results and Discussions

Figures 4a-h indicate the microstructure of 30HGSA steel before and after volumetric and surface heat treatment. Metallographic analysis showed that 30HGSA steel in the initial state consists of a ferrite-pearlite structure. A fine-needle martensitic structure is formed after electrolyte-plasma hardening. Coarse-acicular martensite is formed after volumetric quenching with cooling in water and oil. No significant changes are observed in the structure of quenched samples after annealing.

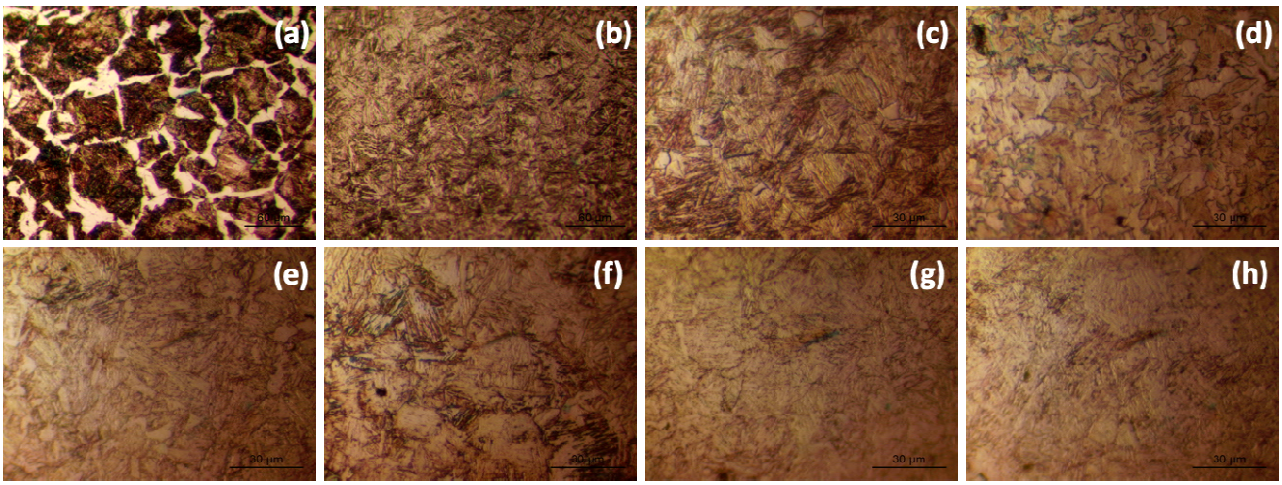


Figure 4. Microstructure of 30HGSA steel samples: Q0-30HGSA (a), EPH-30HGSA (b), Q1-30HGSA (c), Q2-30HGSA (d), Q1A1-30HGSA (e), Q2A1-30HGSA (f), Q1A2-30HGSA (g), Q2A2-30HGSA (h)

The phase composition of the samples was investigated before and after volumetric and surface heat treatment. Figures 5a-h indicate X-ray diffraction patterns of 30HGSA steel samples. X-ray structural analysis demonstrated that in the initial state 30HGSA steel consists of the α-phase. After electrolyte-plasma hardening and volumetric quenching with cooling in oil, the diffractograms of the samples, along with the α-phase, exhibit a reflection (121) of cementite. Only the lines of the α-phase are present on the diffraction patterns of the samples that have passed quenching with cooling in water and quenching with subse-

quent annealing. In this case, in all quenched samples, the diffraction patterns show broadening of the interference lines of the-phase. Broadening of the α-phase interference lines is associated with an increase in the dislocation density, the formation of martensite and is mainly determined by the tetragonality of martensite [12-14]. The largest broadening of the α-phase peaks is observed for the Q1-30HGSA sample, which indicates significant internal stresses due to the high cooling rate. In the samples annealed after quenching, such a large broadening is not observed.

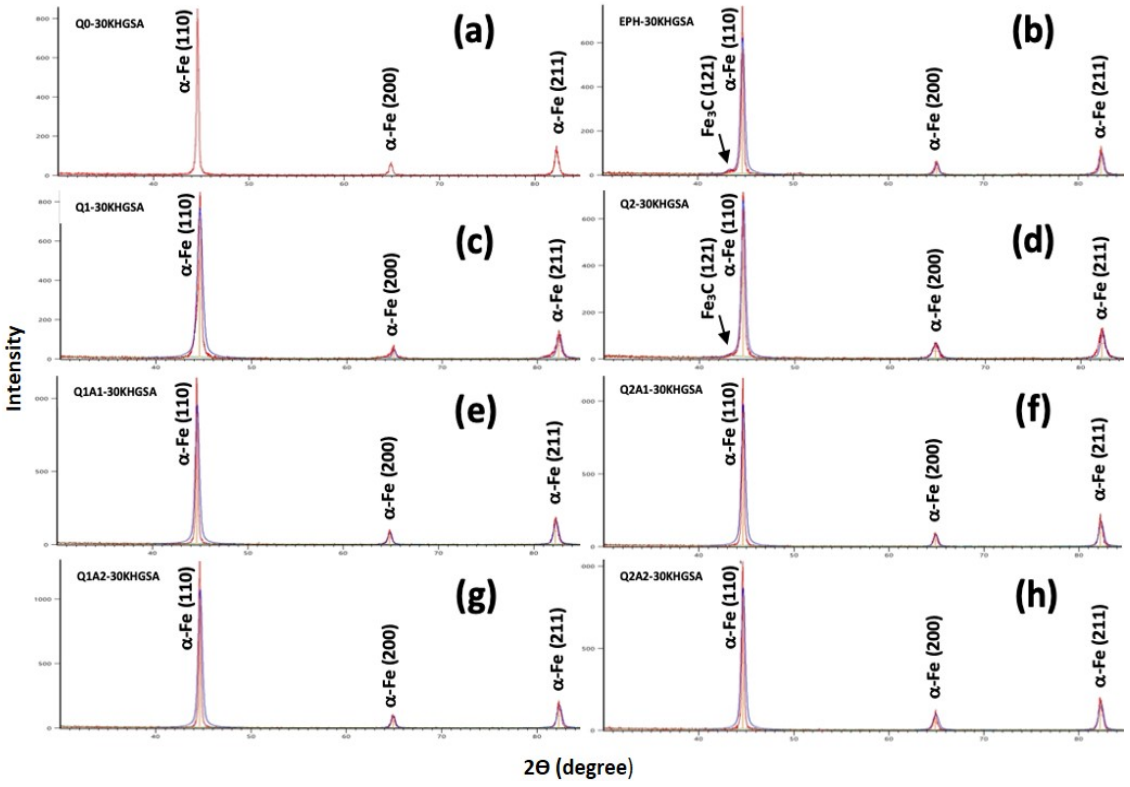


Figure 5. X-ray diffraction patterns of steel 30HGSA: Q0-30HGSA (a), EPH-30HGSA (b), Q1-30HGSA (c), Q2-30HGSA (d), Q1A1-30HGSA (e), Q2A1-30HGSA (f), Q1A2-30HGSA (g), Q2A2-30HGSA (h)

One of the most important properties of the surface layer, which strongly depends on the cooling rate during quenching, is hardness. Therefore, we have studied the changes in the microhardness of 30HGSA steel depending on the mode of heat treatment. Figure 6 represents a histogram of the microhardness of 30HGSA steel before and after volumetric and surface heat treatment. It was identified that the microhardness of steel samples increases after volumetric and surface heat treatment. At the same time, the microhardness increased to 400-460 HV, and subsequent annealing led to a decrease in hardness to 330-360 HV after volume quenching. The decrease in the hardness of quenched samples after annealing is associated with the removal of the internal stress formed during cooling at a high rate.

The maximum increase in hardness is observed in samples treated by plasma electrolyte hardening. An increase in hardness up to 2 times during electrolyte-plasma hardening is associated with the formation of fine-needle martensite. There is no need for annealing due to the small thickness of the hardened layer for steels that have undergone electrolytic-plasma hardening. Only the surface layer with a thickness of 1-2 microns is hardened during electrolytic-plasma hardening, and the base remains viscous. In this case, the hardened layer smoothly passes to the base of the material. Due to the formation of a transition zone – a heat-affected zone, the formed internal stress on the modified layer does not lead to the destruction of the steel material and the appearance of cracks in it.

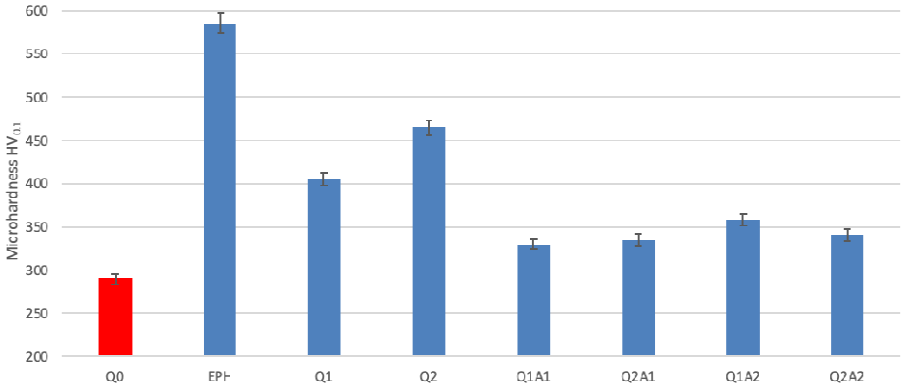


Figure 6. Microhardness of 30HGSA steel before and after surface and volume hardening

Figure 7 designates the results of tribological tests of 30HGSA steel samples according to the "ball-disk" scheme [15]. The wear resistance of the samples was characterized by the coefficient of friction and the volume of wear of the samples. Figure 7a shows the curves of the friction coefficient of the samples before and after treatment. It can be seen from the figure that the samples that have undergone electrolytic-plasma surface hardening and volumetric quenching with cooling in oil have a low coefficient of friction in comparison with the samples that have undergone quenching with cooling in water. In this case, annealing with cooling in oil of the quenched sample leads to a decrease in the coefficient of friction. Specimens quenched with cooling in water and annealing with cooling in water have high values of the coefficient of friction in comparison with the original specimen. Apparently, since steel quenching with cooling in water is accompanied by the formation of structural stresses in the steel this leads to the formation of microcracks under the action of dynamic loads.

Figure 7b represents the results of changes in the volume of wear of the samples before and after treatment. The data on the volume of wear of the samples correlates well with the data on the coefficient of friction of the samples. Samples EPH, Q2 and Q2A2 illustrated low wear volume compared to other samples. The rest of the treated samples showed a high amount of wear compared to the original sample. The low wear volume and coefficient of friction of samples EPH, Q2 and Q2A2 demostrated high wear resistance in dry friction.

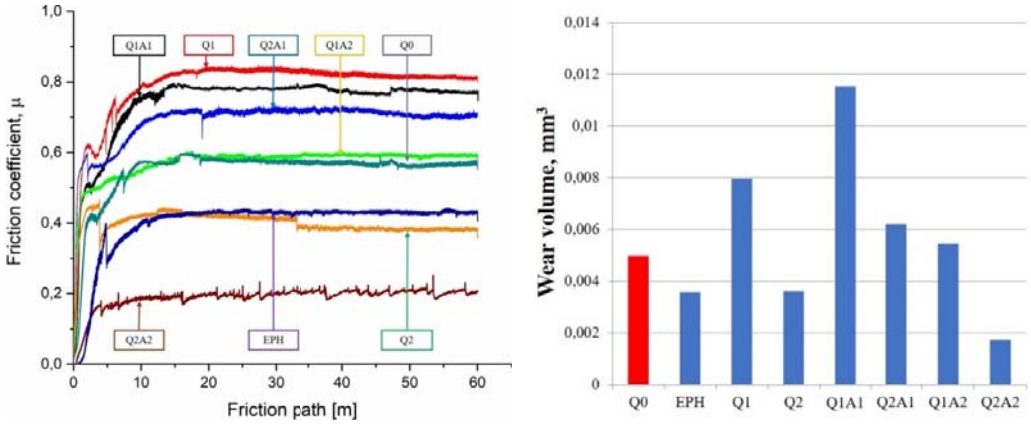


Figure 7. Friction coefficient (a) and wear volume (b) of 30HGSA steel samples before and after surface and volume quenching

Figure 8 demonstrates the results of an abrasive wear test. All samples, except for samples Q1 and Q1A2, showed a low value of weight loss compared to the original sample. At the same time, samples EPH, Q2 and Q2A2 showed high resistance to abrasive wear. The high durability of samples that have undergone electrolyte-plasma surface hardening is associated with the formation of fine-acicular martensite. An increase in the complex of operational properties of steel 30HGSA during electrolytic-plasma hardening is carried out due to saturation of the solid solution with carbon and alloying elements, grain refinement, and an increase in the density of crystal structure defects.

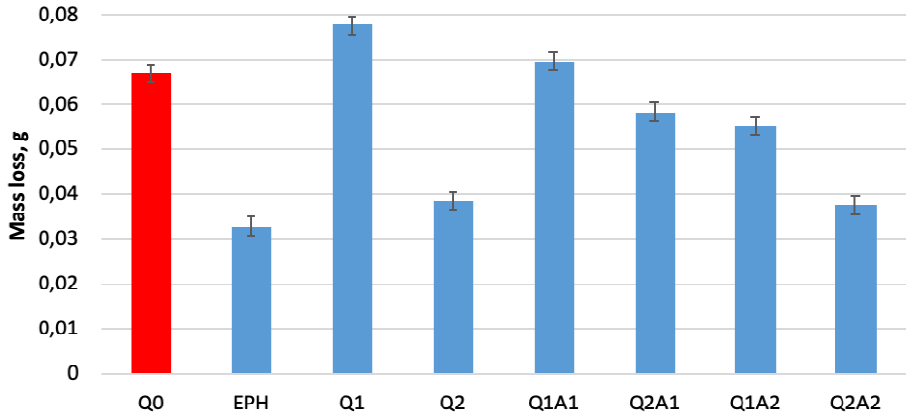


Figure 8. Results of abrasive wear resistance tests of 30HGSA steel samples

Conclusions

It was determined that after electrolytic-plasma hardening, fine-acicular martensite with a small content of cementite is formed on the basis of metallographic and X-ray structural analyses, and coarse-acicular martensite is formed after volume quenching in water and oil. In this case, cementite is observed in the diffractogram after quenching with cooling in water. The cementite dissolves after annealing. No significant changes are observed after annealing in the structure of the samples quenched with cooling in water.

It was analyzed that the microhardness increased to 400-460 HV, and subsequent annealing leads to a decrease in hardness to 330-360 HV after volume quenching. Moreover, due to the formation of fine-acicular martensite the electrolyte-plasma surface hardening leads to an increase in microhardness up to 2 times.

The results of tribological tests of 30HGSA steel specimens before and after heat treatment showed that specimens subjected to electrolytic-plasma surface hardening and volumetric quenching with cooling in oil have a low coefficient of friction in comparison with specimens quenched with cooling in water. It was also determined that samples EPH, Q2 and Q2A2 demonstrated a low wear volume compared to the original sample. Specimens quenched with cooling in water and annealing with cooling in water showed a high volume of wear compared to the original specimen. Apparently, this is due to the fact that quenching and/or annealing of steel with cooling in water is accompanied by the formation of structural stresses in the steel, leading to the formation of microcracks under the action of dynamic loads.

The results of testing samples of steel 30HGSA for resistance to abrasive wear indicated that the samples, except for Q1 and Q1A2, showed a low value of weight loss in comparison with the original sample. At the same time, samples EPH, Q2 and Q2A2 showed high resistance to abrasive wear.

Thus, the tribological properties of parts made of steel 30HGSA can be increased due to the use of heat treatment, including quenching from 900 ºC with cooling in oil, followed by annealing with cooling in oil. Also, surface plasma hardening can be used, which includes heating for 2 seconds followed by cooling in a flowing electrolyte. Electrolytic-plasma surface hardening is a more economical and productive process compared to bulk heat treatment. Concurrently, after electrolytic-plasma surface hardening, the hardness of steel 30HGSA increases by 2 times and tribological properties increase. This is primarily due to the formation of a highly dispersed metastable structure with a higher dislocation density in the surface layer.

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30ХГСА болатының құрылымы мен қасиеттеріне көлемді термиялық өңдеу мен электролитті-плазмалық беткі шынығудың әсері

Мақалада 30ХГСА болатының құрылымдық-фазалық күйлеріне, қаттылығы мен тозуға төзімділігіне, көлемді және беттік термиялық өңдеудің әсерінің салыстырмалы зерттеу нәтижелері келтірілген. Беткі қатаю электролитті-плазмалық әдіспен жүзеге асырылды. Үлгілердің көлемді қатаюын 900°С температураға дейін қыздыру арқылы жүргізілген, содан кейін суда және майда, сондай-ақ, қатаюдан кейінгі үлгілердің бір бөлігі 510°С температурада салқындатылады. Металлографиялық және рентгендік талдау әдістерімен 30ХГСА болат үлгілерінің құрылымдық-фазалық күйлері зерттелді. Микроқаттылықты өлшеу, шар-диск схемасы бойынша трибологиялық сынақтар жүргізілді, сондай-ақ, үлгілердің абразивтік тозуға төзімділігі анықталды. Металлографиялық және рентгенқұрылымдық талдаулар негізінде электролитті-плазмалық шынығудан кейін құрамында аз цементит бар ұсақ инелі мартенсит, ал көлемді шынығудан кейін суда және майда түзілетіні белгілі болды. Көлемді қатаюдан кейін микроқаттылықтың 400-460 HV дейін жоғарылағаны анықталды, содан кейін күйдіру қаттылықтың 330-360 HV дейін төмендеуіне әкеледі. Электролитті-плазмалық беткі қатаю ұсақ инелі мартенситтің пайда болуы есебінен микроқаттылықты 2 есеге дейін арттыруға әкелетіні дәлелденген.

Кілт сөздер: беріктендіру, қыздырып босаңдату, көлемдік шынықтыру, электролитті-плазмалық өңдеу, құрылым, желінугетөзімділік, мартенсит.

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Влияние объемной термической обработки и электролитно-плазменной поверхностной закалки на структуру и свойства стали 30ХГСА

В статье представлены результаты сравнительного изучения влияния объемной и поверхностной термической обработки на структурно-фазовые состояния, твердость и износостойкость стали 30ХГСА. Поверхностная закалка осуществлялась электролитно-плазменным методом. Объемная закалка образцов проведена нагревом до температуры 900°С с последующим охлаждением в воде и в масле, а также

часть образцов после закалки подвергнута отжигу при температуре 510°С. Были изучены структурно-фазовые состояния образцов стали 30ХГСА методами металлографического и рентгеноструктурного анализа. Проведены измерение микротвердости, трибологические испытания по схеме «шар–диск», а также была определена стойкость образцов к абразивному изнашиванию. На основе металлографического и рентгеноструктурного анализа определено, что после электролитно-плазменной закалки формируется мелкоигольчатый мартенсит с небольшим содержанием цементита, а после объемной закалки в воде и в масле формируется крупноигольчатый мартенсит. После объемной закалки микротвердость увеличивалась до 400–460 HV, а последующий отжиг приводил к уменьшению твердости до 330–360HV. Выявлено, что электролитно-плазменная поверхностная закалка приводит к увеличению микротвердости до 2 раз за счет формирования мелкоигольчатого мартенсита.

Ключевые слова: упрочнение, отжиг, объемная закалка, электролитно-плазменная обработка, структура, износостойкость, мартенсит.

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