Influence of electrolytic-plasma hardening modes on structure and hardness of 0.34Cr-1Ni-Mo-Fe steel

B.K. Rakhadilov[1,2], Z.A. Satbayeva[1], R.S. Kozhanova[*,2,3],
D.R. Baizhan[1], M.K. Rakhadilov[2], G.B. Botabayeva[1]

[1]Sarsen Amanzholov East Kazakhstan University, Ust-Kamenogorsk, Kazakhstan

[2]PlasmaScience LLP, Ust-Kamenogorsk, Kazakhstan

[3]Shakarim University of Semey city, Semey, Kazakhstan

E-mail: kozhanovars@yandex.kz

The article presents the results of studying the process of electrolytic-plasma hardening of 0.34Cr-1Ni-Mo-Fe steel by surface hardening, as well as the results of the current-voltage characteristics of the cathodic electrolytic-plasma process depending on the composition of the electrolyte. Temperature-time and special modes of electrolytic-plasma hardening of steel 0.34Cr-1Ni-Mo-Fe were determined. The optimal composition of the electrolyte for electrolytic-plasma hardening has been determined, providing a relatively high heating rate and high hardness of the steel surface. It has been determined that after the electrolytic-plasma hardening, the microhardness of 34KhN1M steel increases 2.9 times due to the formation of fine martensite. In this case, the basis of the material does not change, it consists of a ferrite-pearlite structure.

Keywords: electrolytic-plasma hardening, microhardness, structure, steel, electrolyte.

Introduction

Development of new methods of influencing the surface to give it the necessary operational properties is one of the urgent tasks of modern science and technology. The performance of machines and mechanisms depends on the wear resistance of parts, which often experience large shock loads. For durability, the parts must have high strength and hardness of the surface layer, combined with sufficient

ductility of the core. This can be achieved by using various methods of surface hardening. One of such methods is the electrolytic-plasma treatment of metals [1-3].

Currently, there is a considerable interest in the electrolytic-plasma treatment based on electric discharge phenomena in the "metal-electrolyte" system. Electrolytic-plasma processing method is largely devoid of the disadvantages inherent in traditional methods of metal surface processing, and additionally allows to save material resources. The essence of the process of electrolytic-plasma treatment is as follows. At low voltages in an electrochemical cell containing an aqueous electrolyte solution, a classical electrochemical process is observed. With increasing voltage, intense electrode gas evolution begins, leading to the formation of a near-electrode gas-liquid layer. As the voltage increases, the packing density of gas bubbles in the near-electrode gas-liquid layer increases, and the total cross-sectional area of electrolyte bridges between them decreases. As a result of heat release, the temperature of the electrolyte in these bridges reaches the boiling point. During the transition from bubble boiling to film boiling, a thin (50-100 µm) vapor-gas shell appears around the product immersed in the electrolyte, consisting of water vapor activated by OH^-, H^+ and ions of the electrolyte. Micro-discharges flowing through the vapor-gas shell migrate over the surface of the product and transfer significant portions of energy (hundreds of KJ/mol) to localized areas. A complex of physicochemical processes takes place in the vapor-gas shell: film boiling in an electric field; heat transfer; vapor ionization; movement of electric charges [1-5].

The formation of a stable continuous vapor-gas shell around the active electrode is a prerequisite for the implementation of electrolytic-plasma hardening. The formation of a stable continuous vapor-gas shell is influenced not only by the parameters of the electric circuit, but also by the molecular properties of the liquid, which depend on the composition of the electrolyte and its concentration [6, 7]. In addition, the main indicators of the quality of electrolytic-plasma treatment, which include hardness, thickness of the modified layer and wear resistance, depend on a number of factors: electrolyte composition, current density, voltage, electrolyte temperature, and processing time. Among these factors, the most important role belongs to the composition of the electrolyte.

For surface hardening of steels, aqueous solutions of sodium carbonate are most widely used [8]. As is known [9-11], for the cathodic process, the most optimal component providing a stable discharge is sodium carbonate. In order to prevent the process of decarburization, carbon-containing components such as glycerin, carbamide, etc. are added to the electrolyte.

In this regard, the aim of this work was to select the optimal composition of the electrolyte and technological modes for the electrolytic-plasma hardening of 0.34Cr-1Ni-Mo-Fe steel.

Material and research metod

Structural alloyed 0.34Cr-1Ni-Mo-Fe steel was chosen as the object of research. Table 1 shows the chemical composition of the studied steel.

Table 1.
Chemical composition of steel GOST 4543-71.

Steel name	C	Si	Mn	Ni	S	P	Cr	Cu
34KhN1M	0.3-0.4	0.17-0.37	0.5-0.8	1.3-1.7	below 0.035	below 0.03	1.3-1.7	0.2-0.3

EPH of steel samples was carried out on a unit that was developed and manufactured by the authors at "Surface Engineering and Tribology" research center of S. Amanzholov EKU. EPH process was carried out in an electrolyte from an aqueous solution containing sodium carbonate and carbamide in various concentrations 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 $\approx$ 850-900 $^\circ$C. Cooling was carried out in a flowing electrolyte after turning the voltage off. The heating temperature of the studied sample was measured using a digital multimeter UNIT-33C and chromel-alumel thermocouple (accuracy 2% in range of 40 to 1000 $^\circ$C). Thermocouple was inserted into a hole drilled in a cathode at a distance of 2 mm from heated surface. The layout of the thermocouple is shown in Figure 2.
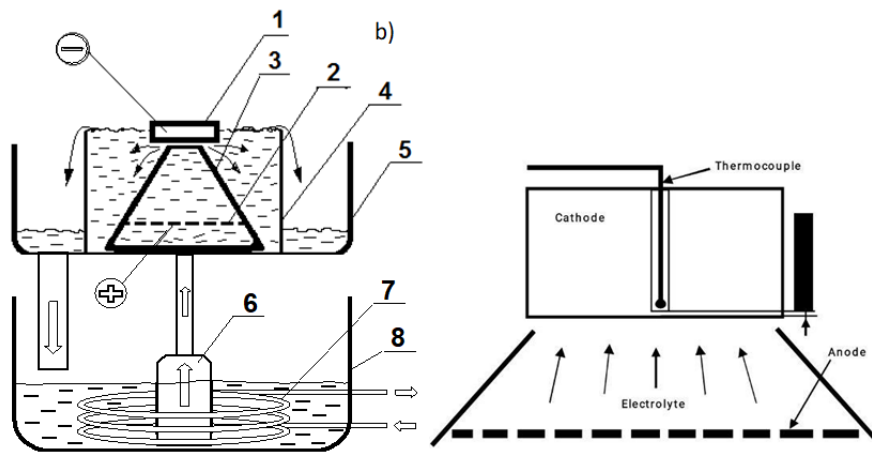

Figure 1. Scheme (b) of UEPOM-M EPT system and representing the position of the thermocouple in a sample during temperature measuring: 1 – processed sample (cathode), 2 – stainless steel anode, 3 – cone-shaped partition, 4 – electrolytic cell, 5 – pallet, 6 – pump, 7 – heat exchanger, 8 – bath with electrolyte.

Experiments to study the energy characteristics were carried out with electrolytes containing carbamide and sodium carbonate with different concentrations (Table 2). In all experiments, the voltage across the electrodes and the average current between them were recorded using dial gauges.

Table 2.
Electrolyte composition.

Electrolyte No.	Sodium carbonate Na_2CO_3, %	Carbamide $(NH_2)_2CO$, %	Water H_2O,%
1	10	10	70
2	20	10	80
3	15	15	70
4	10	20	70
5	20	20	60

The microhardness of the samples was measured by the method of indentation of a diamond indenter on PMT-3 device in accordance with GOST 9450-76, at a load of 100 g and holding under a load of 10 s. The roughness was determined on profilometer of model 130 in accordance with GOST 25142-82.

Results and discussion

Figure 2 shows the current-voltage characteristics of the electrolytic-plasma process in electrolytes containing carbamide and sodium carbonate with different concentrations. It is seen that the current density for all concentrations of electrolyte at the same voltage has approximately the same value, i.e. the magnitude of the current passing through the sample in this concentration range does not strongly depend on the electrolyte concentration. From the type of current-voltage characteristics, it can be seen that with an increase in the concentration of sodium carbonate, the current density increases. It was also found that an increase in carbamide content leads to an increase in current density at low concentrations of sodium carbonate. In addition, at high concentrations of sodium carbonate, carbamide practically does not affect the current density.
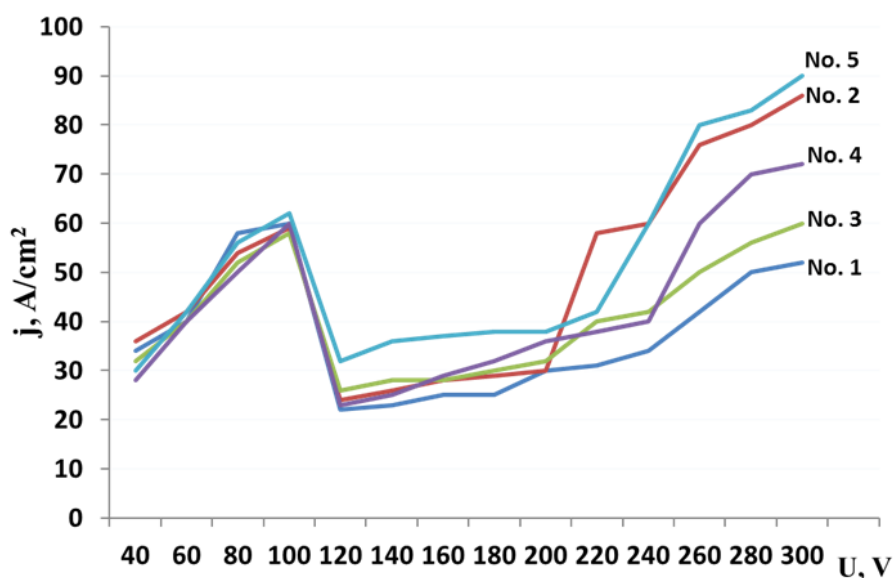

Figure 2. Voltage-current characteristic of the electrolyte-plasma heating process.

The temperature-time dependences of heating were studied for each electrolyte. Figure 3 shows a plot of temperature versus time for the electrolytes under consideration. However, due to the difficulty of accurately determining the temperature during short-term heating, we were unable to reveal the difference in the heating rate of the sample for each electrolyte. At the same time, all considered electrolytes showed a high heating rate - 450-500 °C.
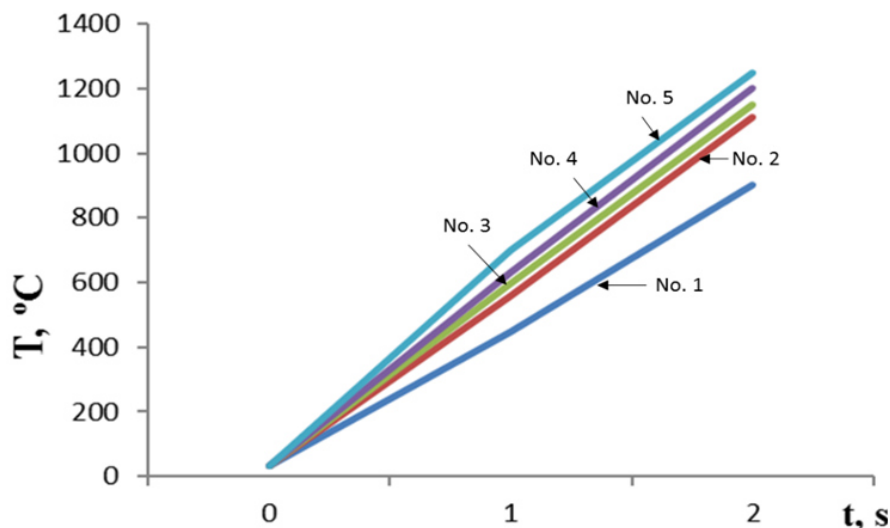

Figure 3. Dependence of the heating temperature of the sample on the time of the electrolytic-plasma treatment.

Hardness is one of the most important properties of the surface layer, which strongly depends on the rate of heating and cooling during surface hardening. Therefore, we studied the changes in the microhardness of 34KhN1M steel depending on the composition of the electrolyte. Figure 4 shows the dependence of the microhardness of 34KhN1M steel before and after electrolytic-plasma hardening in various electrolytes. EPH was carried out by exposure to electrolytic plasma for 2 s, followed by cooling in a flowing electrolyte. The electrolyte temperature was ≈ 40 °C. High hardness is observed in samples treated in electrolytes containing 20% sodium carbonate and 10% carbamide. In addition, the samples processed in electrolytes containing 20% carbamide and 10% sodium carbonate showed the lowest hardness.

Figure 5 shows the dependence of the microhardness of 34KhN1M steel on the time of exposure to plasma in an electrolyte containing 20% sodium carbonate and 10% carbamide. Figure 5 shows that heating for 1 sec practically does not affect the microhardness of the steel. Electrolytic-plasma exposure with a duration of 2 seconds leads to an increase in microhardness up to 7 GPa. An increase in the duration of exposure to 3 seconds will lead to a decrease in microhardness, which is associated with partial melting of the surface layer of steel.

Changes in the roughness of steel samples during electrolytic-plasma hardening were studied depending on the duration of the electrolytic-plasma process. Figure 6 shows the dependence of roughness on the duration of the electrolytic-plasma treatment. It is seen that with an increase in the heating duration, the roughness parameter increases. At the same time, after electrolytic-plasma hardening at the optimal mode (heating duration 2 sec), the roughness parameter is
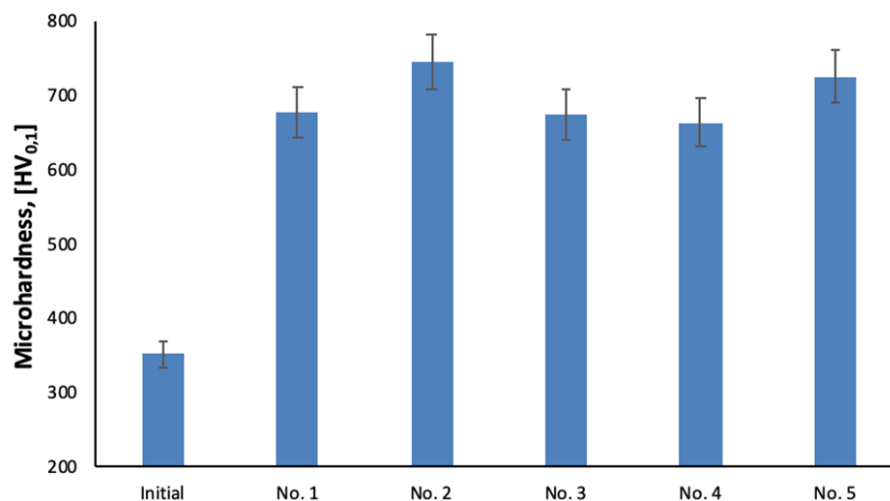
Figure 4. Dependence of the microhardness of 34KhN1M steel on the electrolyte composition.
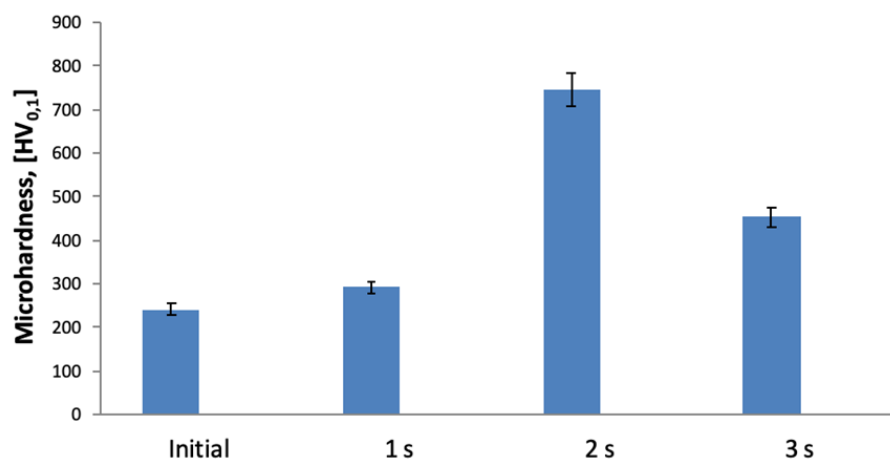

Figure 5. Dependence of microhardness of 34KhN1M steel on treatment time.

0.45 µm, which is acceptable for many machine parts and mechanisms. Thus, the proposed EPH method can be used as a finishing treatment of steel products.
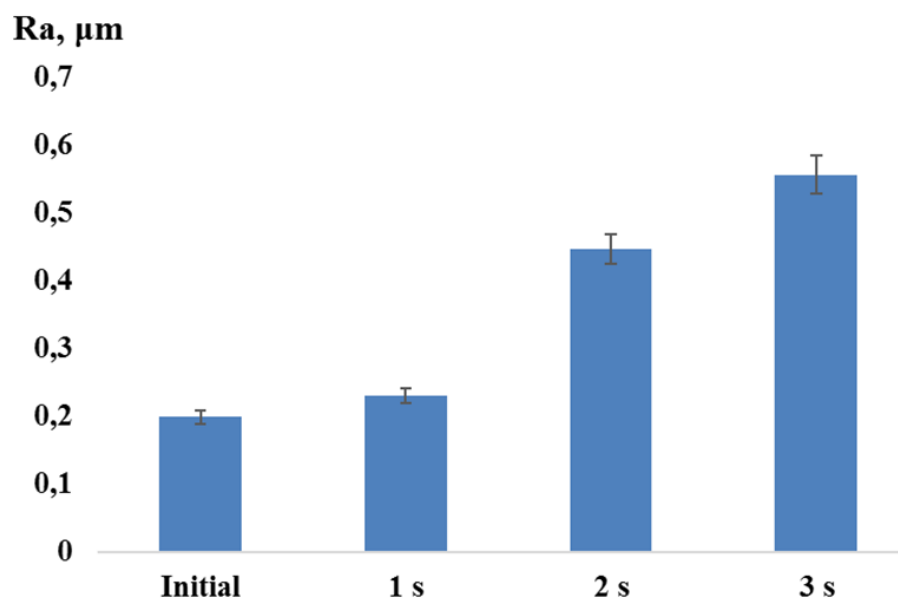

Figure 6. Dependence of roughness on the time of exposure to plasma.

Heating of the electrolyte is a consequence of the action of the current, as well as radiation. The main energy costs are spent on the evaporation and heating of the electrolyte (formation of a plasma layer) and heating the surface of the product with electric discharges. The discharges have the form of micro-arc discharges evenly distributed over the processing area [7].

With periodic switching on of high voltage electric potential (320 V) and low (180 V), a periodic increase and decrease in the heating rate is observed, which makes it possible to increase the time and obtain a thicker heated layer. Connecting an electric potential at the time of cooling the surface of the product allows reducing the cooling rate, and creates the ability to harden products that are made of an alloy with a high carbon content.

By periodically changing the heating power density, hardened layers with a thickness of 0.5 mm, 2 mm, 4 mm, 7 mm, 8 mm and 10 mm can be obtained. As can be seen from Figure 7, heating at a high voltage of 320 V without switching low voltage makes it possible to obtain a hardened layer with a thickness of 0.5-0.6 microns. In order to obtain a hardened layer with a thickness of 4 mm, it is necessary to heat for 25 seconds with periodic switching on of high voltage U1=320 V for 1 second and low voltage U2=180 V for 4 seconds. Total heating for 50 seconds provides a hardened layer thickness of 10 mm. The experiment was carried out on a flat sample of 34KhN1M steel, which had a thickness of 50 mm. Heating and cooling was carried out with a heater having an outlet nozzle diameter of 20 mm. The electrolyte contained 20% sodium carbonate and 10% carbamide.

x - U_v=320 V, t=2 sec; $\Diamond$ - U_v=320 V, U_n =180 V, t=7 sec; $+$ - U_v=320 V, U_n =180 V, t=25 sec; $\square$ - U_v=320 V, U_n =180 V, t=50 sec; o - U_v=320 V, U_n =180 V, t=60 sec; Δ - U_v=320 V, U_n =180 V, t=70 sec
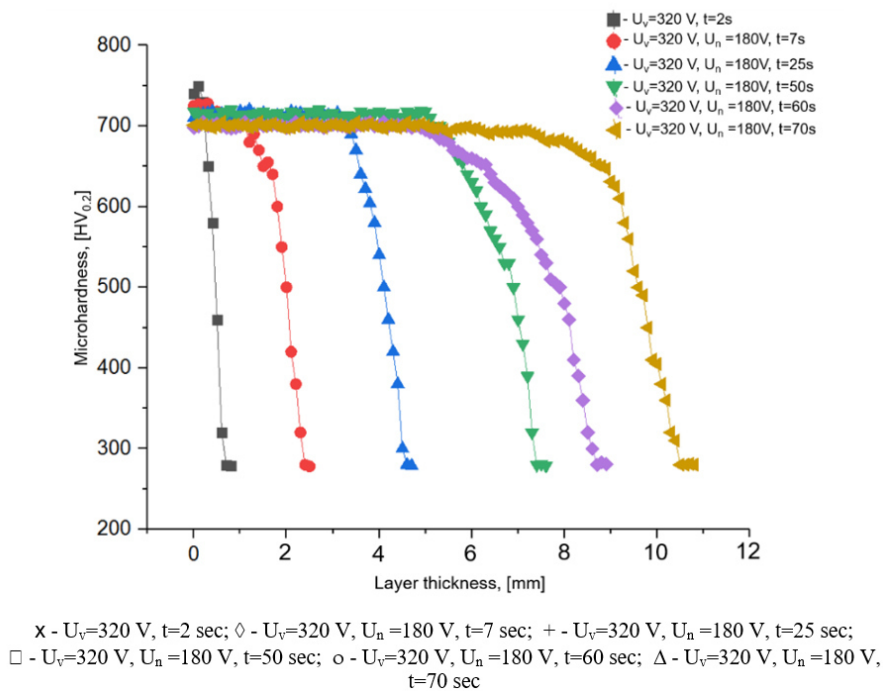
Figure 7. Hardness of the hardened layer on the surface of 34KhN1M steel depending on the time - t of heating and voltage of the electric potential.

Analysis of the results of the experiment (Figure 7) shows that the alternation of switching on the electric potential at a voltage of U1=320 V and U2=180 V

provides heating of the product surface to a depth of 10 mm. In this case, the maximum hardness of the surface layer (750 HV) practically does not depend on the thickness of the hardened layer. The hardness of the hardened layer of the product gradually decreases from the maximum (750 HV) to the hardness of the base (280-300 HV) and, as a rule, does not depend on the heating time. A periodic change in the electric field strength between the surfaces of the liquid electrode and the product changes the power density of the surface heating, which ensures the control of the electrolytic-plasma heating and the creation of the necessary thermal conditions for the formation of quenching structures.

Figure 8 shows fragments of the microstructure obtained using a scanning electron microscope of a transverse section of 0.34C-1Cr-1Ni-1Mo-Fe steel at REN, where the zoning of the structure characteristic of surface hardening is observed. REN steel resulted in modification of the surface layer of the sample. The layer structure changes with distance from the sample surface. It can be seen from the figure that a modified surface layer consisting of martensite is well distinguished on the transverse microsection. The thickness of the modified layer is ≈ 1.7-2 mm.
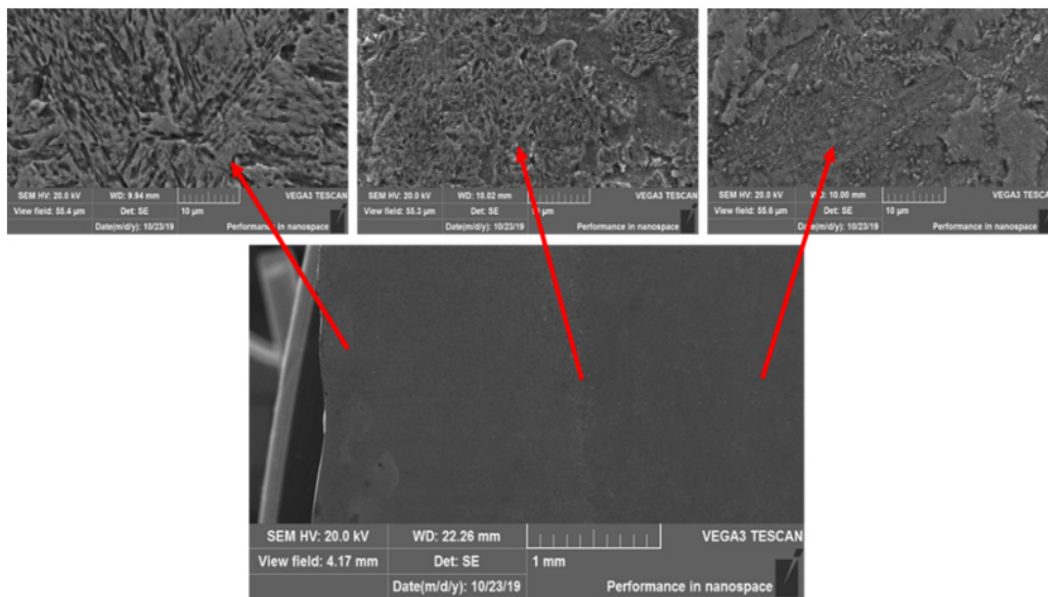

Figure 8. Microstructure of 0.34C-1Cr-1Ni-1Mo-Fe steel cross-section, treated at U_v =320 V, U_n =180V, t =7sec.

Conclusion

1. The optimal composition of the electrolyte for EPH of 34KhN1M steel, containing 20% sodium carbonate and 10% carbamide, which contributes to an increase in the microhardness of steel 34KhN1M by 2.9 times during the processing time of 2 s, has been determined. Thus, the main advantage of EPH is the possibility of obtaining a modified layer on the steel surface with increased microhardness. In this case, the basis of the material does not change, it consists of a ferrite-pearlite structure. The presence of carbide particles in the surface layers has a positive

effect on the performance properties of parts, since small carbides keep products from abrasion.

2. Experiments have shown that, depending on the technological conditions, it is possible to obtain hardened layers on the surface of the product, which have a thickness of 0.5 to 10 mm and a hardness of up to 750 HV. The placement of thermally treated layers on the surface of the product depends on the speed, the trajectory of movement of the electrolyte heaters relative to the surface to be hardened and the design features of the heaters themselves.

3. Regulation of the structural-phase state and thickness of the modified layer by varying the heating time and temperature makes it possible to implement optimal technological modes for obtaining various options for the physical and mechanical properties of steel.

Acknowledgments

References

[1] I.V. Suminov et al., in 2 volumes - M: Technosphere **1** (2011) 464. (in Russian)
[2] A.L. Yerokhin et al., Surface and Coatings Technology **122** (1999) 73-93.
[3] P.N. Belkin, Electronic processing of materials **46**(6) (2010) 29-41. (in Russian)
[4] V.S. Niss et al., Surface Engineering. New powder composite materials. Welding. Part 2. Materials of the Intern. symposium, GNPO PM NAS Belarus (2011) 70-73. [in Russian]
[5] M. Skakov et al., Bulletin of KazNTU **3**(103) 65-71.
[6] V.N. Duradji, A.S. Parsadanyan, Chisinau: Shtiintsa (1988) 216. (in Russian)
[7] Y.N. Tyurin, A.D. Pogrebnyak, Surface and Coatings Technology **142-144** (2001) 293-299.
[8] S.A. Kusmanov et al., Surf. Coat. Technol. **269** (2015) 308-313.
[9] B. Rakhadilov et al., IOP Conference Series: Materials Science and Engineering **142**(1) 012028.
[10] L.G. Zhurerova et al., Journal of Materials Research and Technology (2020) 78-85.
[11] E.V. Parfenov et al., Surface & Coatings Technology **269** (2015) 2-22.