Microstructural Features and Tribological Behaviour of Low-Alloyed Steel Modified by High-Energy Plasma Pulse

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The purpose of the work is to study the microstructure and tribological properties of modified layers obtained on the surface of low-alloyed structural steel 75Mn by the use of high-energy plasma pulse. Subsurface layers were modified using an electrothermal axial plasma accelerator with arc discharge voltage up to 4 kV and discharge current up to 4 kA producing the plasma flux with the power density in the range of (1.4-1.75)·10^9 W/m^2. The microstructure and properties of the layers were investigated using scanning electron microscopy, XRD, microhardness measurement, and “Ball-on-Disk” wear test. The results showed that a single plasma impulse with power density of 1.4·10^9 W/m^2 forms on the surface a modified layer of 13-15 μm thick with average microhardness of 985 HV consisting of ultrafine-grained martensite and 15.9 vol. % of retained austenite. Increasing power density to 1.75·10^9 W/m^2 led to the formation of modified layer of bigger width (22-26 μm) comprising two martensite-austenite sublayers divided by thin (0.3-0.5 μm) austenitic layer. The average microhardness of inner and outer sublayers is 963 HV and 670 HV respectively. The outer sublayer contains higher volume fraction of retained austenite (32 vol. %). It was found that plasma treatment leads to enrichment of the modified layer with plasma-transferred carbon which up to 1.4 wt. %. Plasma modification improves the tribological properties of 75Mn steel, that manifests in increasing wear resistance by 18-90 % and stabilizing the coefficient of friction during the wear testing. The wear process of modified surfaces takes place according to abrasive mechanism with groove formation without intense surface oxidation. The surface deterioration occurs due to multiple deformation of relief elements with detachment of highly deformed microchips.

Keywords: Pulsed-plasma treatment, Modification, Microstructure, Wear resistance, Friction coefficient.

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1. INTRODUCTION

Modification of near-surface layers of metal products is aimed at changing their microstructural state and (in some cases) chemical composition [1]. Modified state is obtained by applying various technologies of surface engineering [2-4], including pulsed-plasma treatment (PPT). As a result of PPT, the surface acquires certain functional properties, which ultimately increase the mechanical and operational behaviour of metal products [5].

Colliding of plasma clot with a metal surface leads to its rapid heating/cooling, which causes phase-structural changes in the near-surface layers to a depth of 25-50 μm [6, 7]. The power of the pulse greatly affects the heating depth and the maximum temperature, which may exceed the metal melting point. In the case of the treatment of steel surfaces, this leads to the formation of a martensitic structure, which is characterized by small sizes of martensite crystals, increased defect density and increased distortion of the crystal lattice. Such a structure has improved hardness and resistance to fracture as compared with bulk thermal treatment [8]. The shock wave from the plasma pulse provides deformation strengthening combined with the implantation of plasma components (atoms, ions) deep into the surface [9].

Different plasma sources are used for pulsed-plasma treatment [1, 5, 8]. They include the electro-thermal axial plasma accelerator (EAPA), the construction and principle of which are described in detail in the papers [10, 11]. The advantages of EAPA are the simplicity of the design and the possibility of processing under atmospheric pressure in air. In EAPA, plasma is formed by the products of evaporation of the electrodes and the dielectric chamber walls. Also, plasma flux carries microdroplets taken from the surface of metal electrodes. This allows the formation of composite coatings, varying with electrodes' material and with PPT mode depending on the electrodes' melting temperature. The microstructure and mechanical properties of Cr-W-Fe-C-B coatings produced by EAPA are described in paper [12]. In articles [6, 7], the time/temperature conditions of modifying low-alloyed steel under PPT are outlined. At the same time, the tribological characteristics of pulsed-plasma modified steel surfaces are insufficiently studied, which does not allow to fully estimate the advantages of PPT over other surface-strengthening technologies. In this regard, the object of the present work is investigating the microstructure and wear resistance of steel 75Mn modified by plasma pulses of different power density.

2. MATERIALS AND METHODS

The specimens of hot-rolled steel 75Mn (0.75 wt. % C, 0.91 wt. % Mn, 0.28 wt. % Si) in sizes of 10 × 10 × 20 mm were subjected to pulsed-plasma treatment. PPT was performed using an EAPA consisting of an array of PTF-6-0.5/10U1 and an electrical circuit including a capacitive energy storage device of 1.5 mF capacity [13]. Pulsed arc discharge with a current of 4 kA and duration of ~ 1.5 ms was initiated be-

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tween the electrodes in a narrow dielectric channel that increased the pressure inside to 100-150 atm. This provided pulsed injection of a plasma clot in the direction of the treated surface.

Pulsed-plasma treatment was performed under the following parameters: the charge voltage of the energy storage device, which is applied to the electrodes, is up to 4.0 kV; the distance between electrodes inside EAPA is 50 mm; the distance from EAPA edge to the specimen surface is 50 mm; the number of pulses is one. The axial electrode was a tungsten rod of 3 mm in diameter. PPT was conducted in two modes differed in power density of the heat flux (q_0) (mode I: $q_0 = 1.4 \cdot 10^9$ W/m^2, mode II: $q_0 = 1.75 \cdot 10^9$ W/m^2). Power density of plasma flux varied by the change in discharge voltage. As shown in [6], mode I resulted in temperature of 1400 ^{o}C on the surface, while mode II led to 1680 ^{o}C with surface melting.

The microstructure of the specimens was examined using a scanning electron microscope JSM-6510 LV (JEOL). To detect the microstructure, chemical etching with a 4 vol. % nital was applied. Microhardness was measured with the NOVOTEST TC-MKB micrometer at 25 g load. The X-ray diffraction analysis was performed with IV Pro (Rigaku) diffractometer in CuKα-radiation. The volume fraction of austenite and carbon content in austenite was calculated according to [14]. The tribological tests with the recording of the friction coefficient were performed on a tribometer (CSM Instruments) according to the "Ball-on-Disk" scheme at room temperature and humidity of 70 %. The attached normal load was 5 N. The specimen served as a "Disk", while the counter body was a corundum ball of 2.97 mm diameter, which made circular movements with a radius of 6 mm at a speed of 0.1 m/s. The total friction distance was 200 m. After each 50 m, the specimen was cleaned by alcohol and weighed on electronic scales with an accuracy of 0.0001 g.

3. RESULTS AND DISCUSSION

3.1 Microstructure of Modified Layers

Fig. 1 presents the microstructure of near-surface layers treated by different PPT modes. As can be seen from Fig. 1a, a modified layer with a thickness of 13-15 μm is clearly identified on the surface of the specimen treated by the mode I. The layer consists of ultrafine-grained martensite, whose structure is detected only with higher magnification (shown in the upper right corner of Fig. 1a). The length of the martensite needles is 0.6-1.1 μm. This indicates that martensite was obtained from ultra-fine austenitic grains formed under super-fast ($4.5 \cdot 10^6$ K/s [6]) heating caused by plasma pulse. The average microhardness of the modified layer is 985 HV.

At PPT under mode II, a modified layer with a total thickness of 22-26 μm (Fig. 1b) was gained on the surface consisting of two sublayers. On the very surface, a sublayer (1) with a thickness of 4-6 μm having austenite-martensitic structure appeared: martensite (dark) with needle length up to 3.3 μm is seen on light austenitic background. The average microhardness of sublayer (1) is 670 HV. Beneath sublayer (1), there is sublayer (2) of 16.5-20 μm thick having finer martensite with needle length up to 2.5 μm. Its average microhardness is 963 HV. Sublayers (1) and (2) are separated by austenitic layer (3) of 0.3-0.5 μm thick. For the reference, the microstructure of non-treated specimen is fine pearlite with average microhardness of 235 HV.

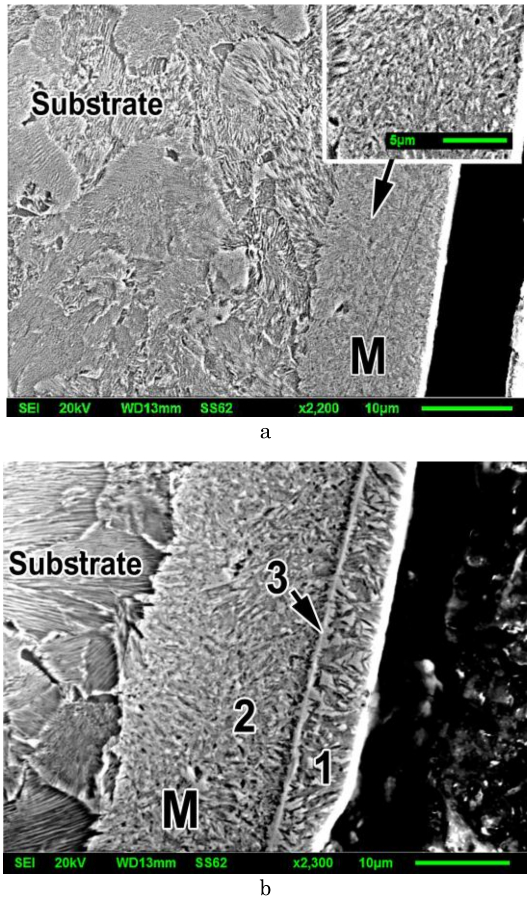


Fig. 1 – The microstructure of modified layer (M) after PPT under mode I (a) and mode II (b)

The phase status of modified layers was determined by X-ray diffraction, the results of which are shown in Fig. 2. Analysis of the XRD patterns showed that non-treated specimen consists mainly of α-iron (ferrite) and, to a lesser extent, of carbide $(Fe, Mn)_3C$. Diffraction peaks related to γ-iron (austenite) were not found on the diffractogram. The ferrite peaks are narrow enough, indicating neither significant distortion of the crystal lattice nor increased amount of the crystalline defects.

After PPT under mode I, the thickening of α-Fe diffraction peaks occurred, reflecting a significant distortion of the crystal lattice due to γ-Fe $\rightarrow$ α-Fe displacive phase transformation. In addition, the peaks (200), (220) and (311) belonging to γ-Fe appeared. This revealed that some austenite retained in the structure during martensite transformation. After PPT under mode II, there was a significant increase in the intensity of austenitic peaks, indicating an increase in volume fraction of retained austenite. Calculations showed that the amount of retained austenite in modified layer is 15.9 vol. % (mode I) and 32.0 vol. % (mode II). Since the thickness of sublayers (1) in Fig. 1b is less than 6 μm, it is obvious that higher amount of austenite (mode II) is associated exactly with this sublayer.

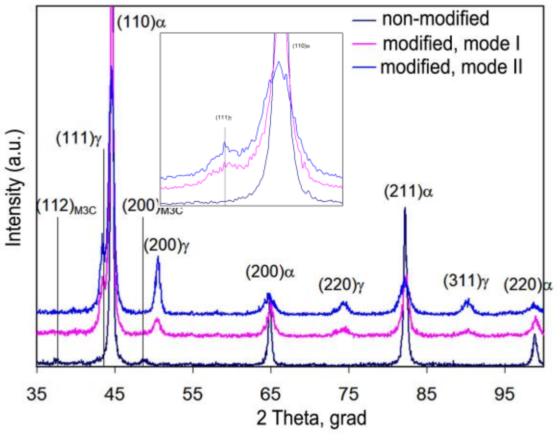


Fig. 2 – XRD-patterns of steel 75Mn in the initial state and after PPT modification under different modes

A question arises about the reason for the difference in the amount of retained austenite in modified specimens, processed under different regimes. The increase in the fraction of austenite can only be explained by plasma-induced enrichment of near-surface layer with carbon atoms. It is known that it is carbon, which most intensively reduces the temperature of the start of martensitic transformation (*Ms*), thus causing an increased volume fraction of retained austenite in quenched steels. The source of carbon atoms could be the inner walls of EAPA discharge chamber, made of paper-reinforced bakelite. Paper and bakelite both consist of carbon, hydrogen and oxygen. During the electric discharge inside the chamber, the paper-bakelite composite is evaporated releasing carbon atoms and ions into the plasma flux. Plasma transfers carbon to the specimen with further implanting into its surface. The XRD pattern-based calculations showed that the carbon content of austenite is 0.94 wt. % for mode I and 1.07 wt. % for mode II, which is 25-40 % higher than total carbon content in steel 75Mn.

Since mode II, according to [6], is accompanied by the surface melting, a more significant saturation of the liquid metal with plasma-transferred carbon atoms may occur. According to this logic, sublayers (1) and (3) with the increased fraction of austenite (Fig. 1b) were melted during the PPT; their thickness (4-6.5 μm) is quite close to the estimated depth of melting (10 μm [6]). Indirect confirmation of this assumption is the increased length of martensitic needles in sublayer (1); this could be due to the growth of austenitic grains when crystallized directly from the liquid phase.

A thin (0.3-0.5 μm) austenitic layer (3) is formed between sublayers (1) and (2). It can be assumed that carbon content in this layer reaches a maximum, thus its Ms does not exceed ambient temperature. *Ms* temperature can be calculated using Popov's formula as: $Ms\,(^{o}C) = 520 - 320\,[C] - 50\,[Mn] - 30\,[Cr] - 20\,[Ni + Mo] - 5\,[Cu + Si]$, where the number in brackets is the content of the chemical element in the steel (in wt. %). Assuming that Ms temperature for the layer (3) is 20 °C, then according to the formula, the carbon content in this layer is 1.41 wt. %. The austenitic layer (3) lies on the inner boundary of the melted layer, therefore uneven distribution of transferred carbon in liquid occurred when plasma pulse interacts with the surface. Due to high kinetic energy, the carbon atoms penetrated into a liquid at a considerable depth, being accumulated at liquid/solid boundary. With the distance from the layer (3) to the surface, the amount of austenite decreases, meaning that the content of dissolved carbon decreases as well. The saturation of the liquid phase with a significant amount of carbon under mode II was facilitated by a higher power density, which promoted EAPA walls evaporation with corresponding enrichment of plasma flux with carbon.

3.2 Tribological Properties of Plasma-Modified Layers

Fig. 3 and Fig. 4 present the results of wear test as the cumulative wear curves and the dynamics of friction coefficient (μ) during the test. It is seen that non-treated specimen is characterized by the highest weight loss (totally $1.33 \cdot 10^{-3}$ g). The specimen modified under mode II has lower weight loss ($1.10 \cdot 10^{-3}$ g), while the best wear performance is shown by the specimen modified under mode I ($0.70 \cdot 10^{-3}$ g). Thus, pulsed-plasma modification without surface melting provided steel 75 Mn with almost doubled increase in wear resistance. In the case of modification with a surface melting, the increase in wear resistance was only 18 %.

The change in the friction coefficient during the test is shown in Fig. 4. An increase in μ value is observed from the very beginning for all three specimens because of gradual deepening of the ball into the surface leading to increase in the contact area. Non-treated specimen is characterized by instability in coefficient μ value. After increasing to 0.54-0.70, μ sharply decreased to 0.36-0.44 after 8 m of friction distance. Subsequently, μ value varied near the value of 0.40, but there was a significant scatter ranging from 0.36-0.43 (32 m) to 0.14-0.75 (17 m). With an increase in the friction distance from 10 m to 30 m, the scatter in μ values decreased, while after about 40 m it again gradually increased.

In the specimen PPT-treated under mode I, coefficient μ performed a more stable dynamics with a lower scatter of values. Coefficient μ increased up to 0.72 in average after about 33 m of friction distance with further stabilization at this level. The scatter of μ values gradually increased from 0.21-0.34 (1 m), stabilizing within 0.55-0.91 after 30 m.

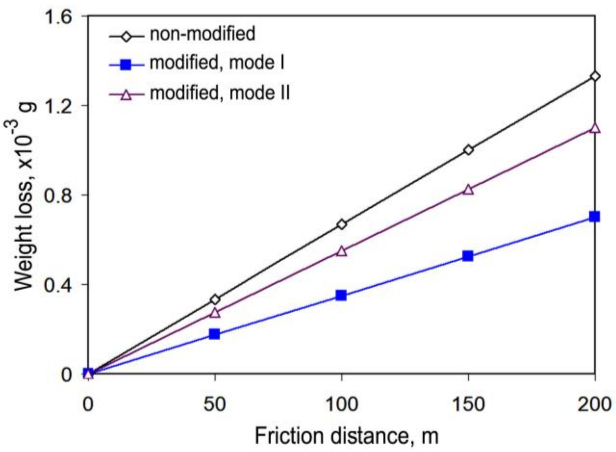
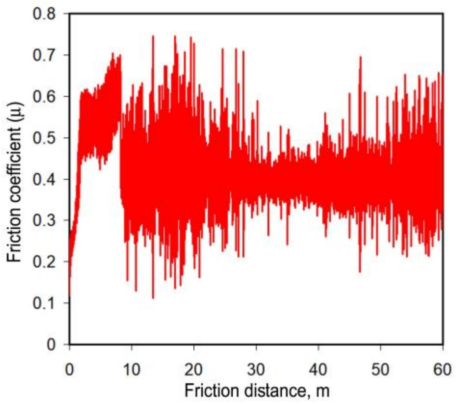


Fig. 3 – Cumulative curves of specimens' weight loss



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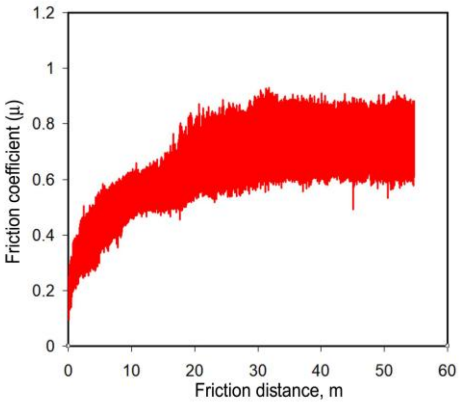

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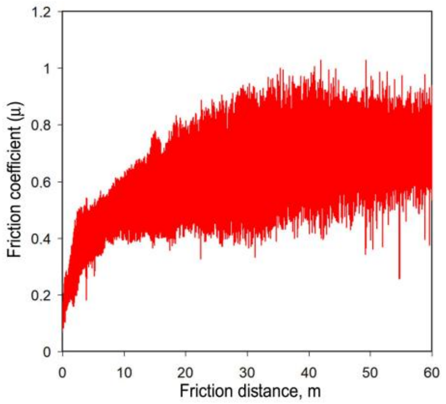

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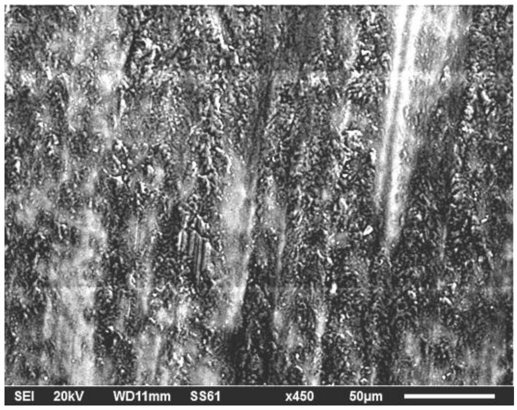
Fig. 4 – Change in friction coefficient during the tests: non-treated specimen (a), PPT mode I (b), PPT mode II (c)

The specimen processed under mode II showed approximately the same dynamics as for previous case, but with increased scatter of μ values (0.25-1.00) for a distance higher than 30 m (the average friction coefficient for this segment is 0.70).

Thus, non-treated specimen is characterized by a minimum level of friction coefficient (0.40 on average) with the largest scatter of μ values during the tests. The reduced average coefficient μ is due to the fact that the soft (unmodified) steel 75Mn was easily deformed in contact with a corundum ball. This led to intense saturation of the surface with oxygen to form thick oxide films that play the role of solid lubricant, reducing the frictional force [15]. Since coarse oxide films are weakly bonded to the surface, their sudden detachment caused "jumps" in μ values. This also led to increased weight loss. Such wear mechanism is confirmed by SEM observation of worn surface. As follows from Fig. 5a, the worn surface of non-treated specimen is densely covered with a large number of oxidation products. Between oxides, there are areas free of oxides that are the places of oxide films detachment.



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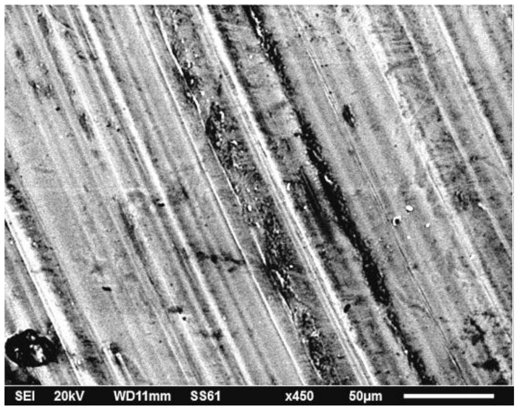

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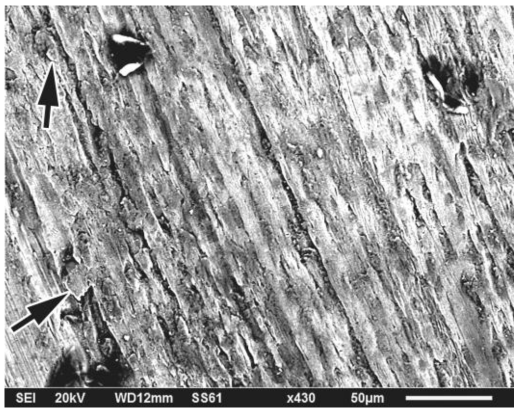

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Fig. 5 – Worn surface of specimens without PPT (a) and plasma-treated under mode I (b) and mode II (c)

The surface of pulsed-plasma treated specimens was deformed with less intensity due to its higher microhardness. This resulted in suppressing the thermal-structural activation with a corresponding decrease in oxidation rate [15]. As a result, lesser amount of oxides appeared on the surface thus increasing the friction force (i.e. coefficient μ). In this case, the wear mechanism changed as compared with unmodified specimen. The hard specimen treated under mode I was worn by the abrasive mechanism with a multiple deformation of relief elements (Fig. 5b). The milder specimen, processed under mode II, was worn with the formation of deeper grooves and intensive deformation of the relief elements with the removal of highly deformed microchips from the surface (shown by the arrows in Fig. 5c).

4. CONCLUSIONS

It is shown that PPT, providing a power density of plasma flux in the range of (1.4-1.75)·10^9 W/m^2, results in modification of 75Mn steel with the formation of subsurface layer of thickness varied from 13-15 μm to 22-26 μm. The modified layers consist of martensite with needle length of 0.6-1.1 μm (mode I) and 1.2-3.3 μm (mode II), and retained austenite. The average microhardness of modified layer (mode II) is 985 HV. The average microhardness of modified layer (mode II) varies from 963 HV (inner sublayer) to 670 HV (outer sublayer). PPT leads to enrichment of modified layer with carbon up to 0.94-1.07 wt. % (locally up to 1.4 wt. %), resulting in increase in volume fraction of retained austenite (15.9-32 vol. %). It is established that PPT improves wear resistance of steel 75Mn by 18-90 % depending on the treatment mode. The wear of modified surfaces is characterized by more stable friction with a lower scatter of friction coefficient, the average value of which is 0.70-0.72. Modified specimens are worn without intense surface oxidation. This process occurs by abrasive mechanism with detachment of highly deformed microchips from the surface.

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Особливості мікроструктури та трибологічні властивості низьколегованої сталі, модифікованої високоенергетичним плазмовим імпульсом

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Метою даної роботи є дослідження мікроструктури і трибологічних властивостей модифікованих шарів, отриманих на поверхні низьколегованої конструкційної сталі 75Г застосуванням високоенергетичного плазмового імпульсу. Модифіковані шари формували за допомогою електротермічного аксіального плазмового прискорювача з напругою розряду до 4 кВ та струмом розряду до 4 кА за поверхневої щільності потужності імпульсу в межах (1.4-1.75)·10^9 Вт/м^2. Мікроструктуру і властивості шарів досліджували з використанням електронної скануючої мікроскопії, рентгеноструктурного аналізу, вимірюванням мікротвердості, а також проведенням випробувань на зношування за схемою «Ball-on-Disk». Результати показали, що однократний плазмовий імпульс з щільністю потужності 1.4·10^9 Вт/м^2 утворює на поверхні сталі модифікований шар товщиною 13-15 мкм зі структурою наддрібнозернистого мартенситу (15.9 % залишкового аустеніту) із середньою мікротвердістю 985 HV. Підвищення щільності потужності до 1.75·10^9 Вт/м^2 привело до формування більш товстого модифікованого шару (22-26 мкм), який складається з двох мартенситно-аустенітних субшарів, розділених тонким (0.3-0.5 мкм) аустенітним шаром. Середня твердість внутрішнього та зовнішнього субшарів становить 963 HV та 670 HV відповідно. Зовнішній субшар вміщує підвищену кількість залишкового аустеніту (32 %). Встановлено, що плазмова обробка призводить до насичення модифікованого шару вуглецем (максимально до 1.4 %), який переноситься плазмовим струменем. Плазмова модифікація підвищує трибологічні властивості сталі 75Г, що проявляється у збільшенні зносостійкості на 18-90 % та стабілізації коефіцієнту тертя впродовж випробувань. Процес зношування модифікованих поверхонь відбувається за абразивним механізмом за відсутності інтенсивного окислення поверхні. Знос поверхні полягає у багатоцикловому деформуванні елементів рельєфу із видаленням перенакльопаних мікрочасток металу.

Ключові слова: Імпульсно-плазмова обробка, Модифікування, Мікроструктура, Зносостійкість, Коефіцієнт тертя.