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PULSE-PLASMA MODIFICATION OF SURFACE OF STEEL HOT DRAWING DIES OF TITANIUM ALLOY PRODUCTS

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The technology of pulse-plasma modification of the working surface of the die of 4Kh5MF1S tool steel (analogues are X40CrMoV5-1 in the EU and 4Cr5MoSiV1 in China) was considered. The mentioned tool is used for stamping billets of titanium VT6 alloy (wt.%: Al — 3.0–6.8; V — 3.5–5.0; Ti — base), which is performed at temperatures of up to 700 °C. The die surface is heated, which leads to its oxidation and diffusion redistribution of alloying elements. Pulse-plasma stamping leads to the formation of elastic-plastic deformations of the surface layer in a tool steel, which in combination with pulsed thermal and electromagnetic effects provides a refinement of the alloy structure and intensifies the diffusion mechanisms of alloying elements. The studies showed that the modified layer (over 80 μm thickness) in 4Kh5MF1S steel, formed in the process of pulse-plasma treatment, contains up to 2.5 % carbon, up to 12 % oxygen and up to 3 % tungsten. In the mentioned layer the presence of nanocrystalline structures with a size of less than 100 nm was revealed. The hardness of the modified layer is more than 700 $HV_{0.025}$. The surface roughness after pulse-plasma treatment did not change. Experience of industrial use of this technology showed that modification of a surface of the die from 4Kh5MF1S steel provided its high serviceability at a deep drawing of products from the heated (to 700 °C) sheet of VT-6 titanium of 3 mm thickness. 11 Ref., 1 Table, 6 Figures.

Keywords: plasma treatment, alloying, tool steels, die, titanium deformation, structuring, wear resistance, serviceability

It is known that nanocrystalline materials have high characteristics of strength and heat resistance [1], but their production in large quantities is currently problematic. The technology of pulse-plasma modification allows modifying the surface layer of a metallic product without its heating. The modification process is accompanied by alloying and nanostructuring of the surface layer. The mechanism of this process is described in the monograph [2].

The working surface of a stamping tool is exposed to the highest loads. Therefore, its modification will significantly increase the capabilities of stamping technology during the deformation of titanium-based alloys. It is proposed to nanostructure and alloy the die surface layer by carbon, oxygen and tungsten. The presence of alloying elements in the surface layer of stamped steel blocks the possibility of oxidation and diffusion processes when the die is heated. Extremely high content of carbon and oxygen in the metal alloy eliminates the chemical and thermal processes of its interaction with titanium, even at high temperatures. Pulse-plasma technology of alloying and nanostructuring of the surface layer is considered in more detail in [3].

The aim of the work is to create a resource-saving effective technology for modifying the surface layer of a stamping tool, which provides its high serviceability in the deformation of chemically active materials and alloys, for example, based on titanium.

Analysis of the work of the tool made of stamped steel after chemical-heat treatment shows [4, 5] that its maximum serviceability is observed after a complex alloying of the surface layer. Thermodiffusion saturation is carried out, as a rule, at a high temperature (900–1100 °C). A long-term heating complicates heat treatment, requires additional power consumption and, furthermore, causes the growth of crystals. Currently, technologies are being developed that allow changing the structural-phase composition of the surface layer to a depth of tens of microns. The technologies of surface laser strengthening, alloying

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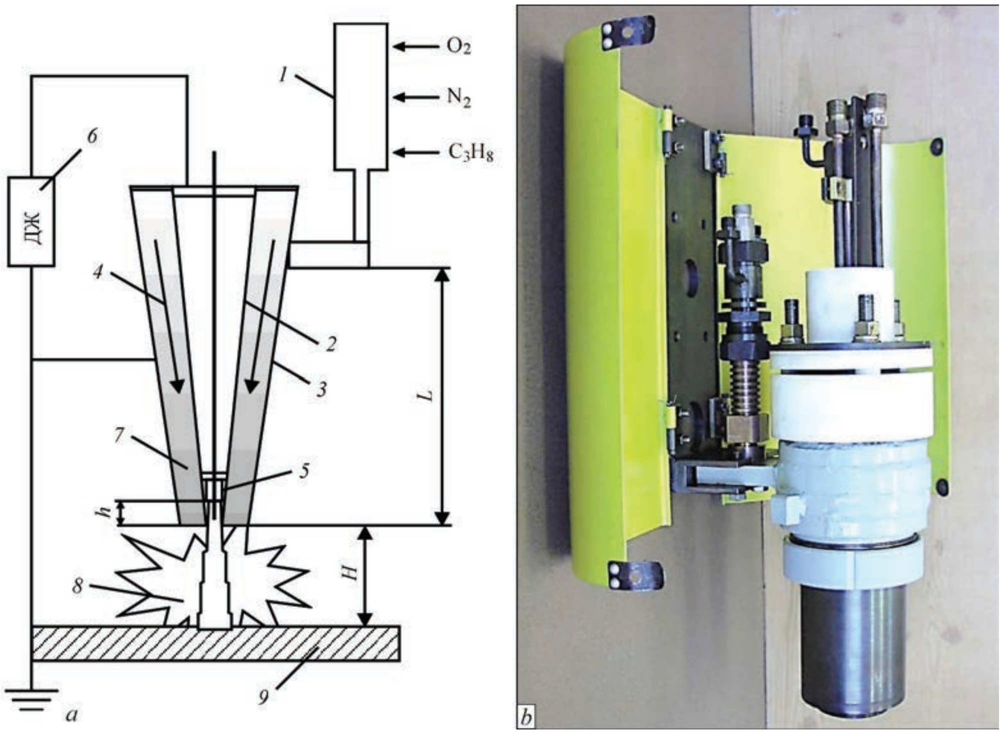


Figure 1. Pulse plasma generator for surface modification: scheme of operating generator (*a*) and appearance of generator on the manipulator arm (*b*)

and nitriding [5, 6], vacuum plasma devices with adjustable atmospheric composition and pressure for surface treatment of tool materials are used [7]. An axial electromagnetic plasma accelerator for surface modification [8] was created, which represents a ballistic plasmatron based on the principle of adiabatic compression of the plasma-forming medium, [9].

The article presents the results of stamped steel modification applying a coaxial pulse-plasma generator [9]. Pulse-plasma treatment provides a rapid heating (10^{-3}–10^{-4} s) of the surface layer of steel with a subsequent intensive cooling. High heating and cooling rates up to 10^7 K/s lead to deformation of the surface layer and, as a consequence, the formation of nanocrystalline structure and high dislocation density. It is possible to introduce different alloying elements into plasma, which in combination with the pulsed electromagnetic influence and elastic-plastic deformation intensifies the mechanisms of their diffusion into the surface layer [10, 11].

To modify the working surface of the dies, a pulse-plasma generator (Figure 1) was used, consisting of a detonation chamber *1*, in which the formation and combustion of a combustible gas mixture (C_3H_8, O_2, N_2) and coaxial electrodes *2, 3* is carried out. A flow of combustion products *4* is formed between the electrodes, having a frequency that can vary within 1–5 Hz. Along the axis of the plasmatron a consumable electrode *5* is located, which evaporates and enriches the plasma with alloying elements.

As a result of detonation combustion of the combustible mixture in the plasma generator, ionized combustion products *7* are formed, which close the *R–L–C* circuit of the power source. In the interelectrode gap of the plasma generator, an electric current flows behind the front of the detonation wave along the cross volume of the gas, the degree of ionization of which subsequently increases. Plasma jet *8* from the generator nozzle flows on the modified surface *9* (cathode). Around the central electrode *2* and the plasma jet 9, an azimuthal magnetic field is formed, which interacts with the current *I* flowing in the interelectrode gap and creates an electromagnetic force that accelerates and focuses the plasma. In addition, during the flow of electric current, Joule heat is released, which due to the expansion of the heated volume of gas also enhances the gas-dynamic component of the plasma, carrying out rapid heating of the surface layer (Figure 2).

The calculated power characteristics of the plasma jets at the output of the plasma generator have a linear dependence on the electric field strength and the length of the interelectrode gap. Thus, at the length of the interelectrode gap $L = 300$ mm and the electric field strength of 400 kV/m, the plasma jet can have a temperature of 25000 K and a velocity of 8 km/s [2].

The plasma jet closes the electrical circuit between the consumable electrode and the surface of a product. A thin layer of material at the end of the consumable electrode is overheated and destroyed by an explosion (transition to a stable state from metastable one), which provides synchronization of the introduction of heated and accelerated elements forming the electrode into the plasma jet. In the zone of inhibition of the plasma jet on the treated surface a layer of shock-compressed plasma and electrode erosion products is formed. The duration of interaction of this layer with the surface of a product is 0.4–0.6 ms, the heat flux varies in the range of $0.3\cdot10^5$–$1.4\cdot10^6$ W/cm^2. The heat flux is regulated by the *R–L–C* parameters of the power source circuit, the distance *H* and the depth of the electrode *h*. The deepening of the electrode mainly affects the diameter of the spot of the plasma contact with the surface of a product (8–25 mm). This in turn allows adjusting the heat flux density at the same pulse energy. In the course of experiments, it was found that under pulse-plasma exposure for 3–5 ms, the products of plasma-chemical synthesis from the interelectrode gap flow and condense on the surface between the pulses. Subsequent plasma pulse melts a thin surface layer, mixing and saturating it with alloying elements. This provides the possibility of additional alloying of the surface layer by preliminary surfacing of alloying elements on the surface of a product.

On the basis of the theoretical analysis of the nonstationary equation of thermal conductivity, the evaluation of heat flows was carried out and change of temperature in time for one pulse of duration $t = 0.6$ ms and power $q = 7.2\cdot10^8$ W/m^2 at different distance from the surface at steel surface treatment was calculated. Calculations showed that heating and cooling rates reach 10^7 K/s, and the temperature gradients across the thickness of the modified layer are $2.5\cdot10^7$ K/m. In the period between the pulses (at a frequency of 2 Hz), the surface layers heated to the melting point have time to cool. A repeated pulse-plasma effect on the surface leads to a periodic deformation of the surface layers, phase hardening and refinement of the structure.

The experiments were performed on a stamped steel of grade 4Kh5MF1S (the analogues are Kh40CrMoV5-1 in the EU and 4Cr5MoSiV1 in China), which is used for manufacture of tools operating in the conditions of long-term heat changes to the temperatures of 630 °C (press stamp, needles for pipe piercing, hammer and press inserts, tool for upsetting of billets, etc.). To increase the heat resistance of the working layer of the tool, it is proposed to increase the content of tungsten, oxygen and carbon in the surface layer, as well as to change its structure to nanocrystalline. The following technological parameters of pulse-plasma treatment were established: inductance of the discharge circuit *L* — 30 μH, capacitance of the capacitor bank *C* — 960 μF and voltage on the coatings of the capacitor bank *U* — 3.2 kV, eroded electrode — *W*. Additional introduction of carbon is carried out by applying graphite coatings to the surface. Consumption of gases: propane — 0.38; oxygen — 1.04; air — 1.13 m^3/h. The potential was supplied from the power source to the product/specimen, which enhanced the plasma effect by switching the power of the electric discharge between the generator electrode (anode) and the modified surface (cathode). The number (5 times) of plasma overlaps on the modified surfaces was established. As is noted above, a change in the value of deepening of the consumable electrode leads to a change in the diameter of the treatment spot, and, accordingly, the heat flux density into a product. For pulse-plasma treatment of specimens and a die a depth at a value h = 25 mm was set. The diameter of the visible treatment area was 18 mm. The specimen and dies were treated at a distance H = 50 mm. This mode provided a fairly high power density, which melted and evaporated the material from the specimen surface.

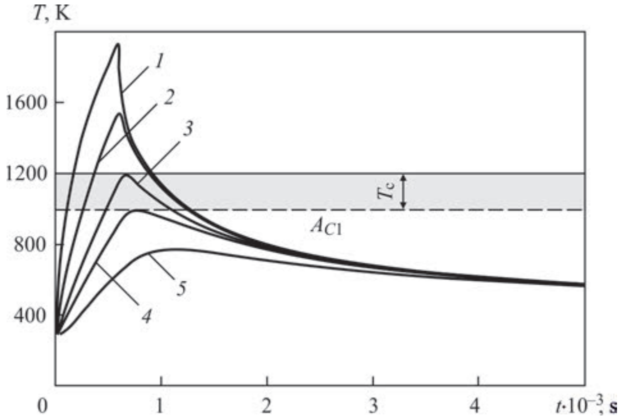
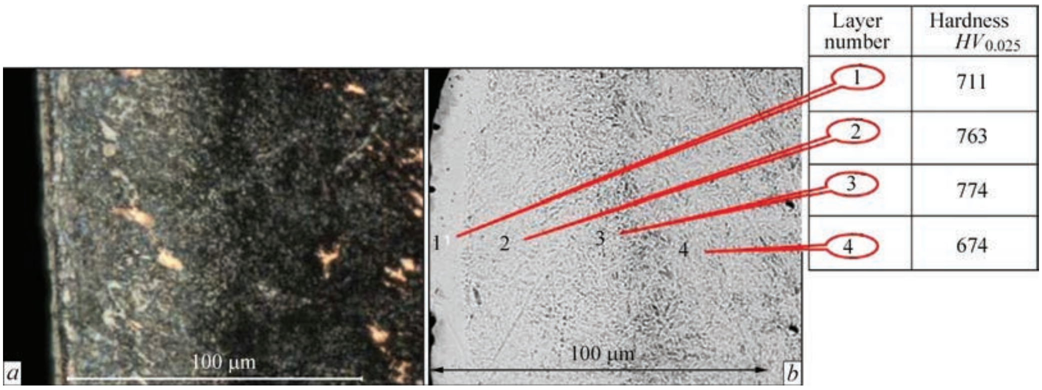


Figure 2. Change in temperature in the specimen layer at the depth: *1* — 0; *2* — 20; *3* — 40; *4* — 60; *5* — 100 μm

Using the methods of optical metallography, visible traces of surface melting were detected on the specimen. Microscopic analysis of the modified surface layer produced with the supply of potential, performed using an electron-ion scanning microscope Quanta 200 3D, showed that on the surface of the specimen a modified layer of up to 100 μm thickness was formed (Figure 3), which has no distinct boundary with the base metal. The hardness of the surface layer increased by 3 times to 774 $HV_{0.025}$, and the hardness of the specimen material at a depth of 80 μm is up to 674 $HV_{0.025}$, which is twice higher than the hardness of the base.

A high power density provided refinement of the structure of the surface layer due to high temperature gradients. However, element-by-element analysis did not show the presence of elements, forming plasma in the layer, which is probably predetermined by a high temperature, evaporation and destruction of a thin



Layer number	Hardness $HV_{0.025}$
1	711
2	763
3	774
4	674

Figure 3. Surface analysis with the use of: *a* — optical inverted microscope OLIMPYS GX 51; *b* —electron-ion scanning microscope Quanta 200 3D

surface layer. The surface of the specimen had roughness and traces of melting, oxidation, carbon deposition and spraying of the material.

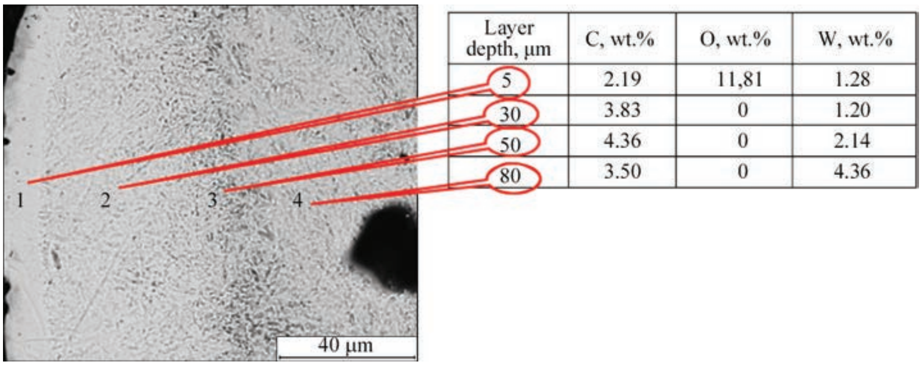
X-ray diffraction analysis confirmed a significant oxidation. On the surface carbon is present, deposited from the graphite coating and condensed from the combustion products. Alloying elements in such conditions are oxidized and removed from the surface together with the destroyed layer of the base metal of the specimen.

Examination of the specimen in a transmission electron microscope equipped with a system of energy-dispersive microanalysis allowed determining that alloying of the surface layer was carried out to a depth of 80 µm (Figure 4). The upper molten layer at a depth of up to 5 µm contains up to 11.81 wt.% of oxygen, 2.19 wt.% of tungsten and 1.28 wt.% of carbon. At a depth of 20–80 µm, the modified layer has 2.3 times (points 2, 3, 4 in Figure 3) increased content of tungsten (3.83, 4.36 and 3.50 wt.%, respectively) and carbon (1.2–4.36 wt.%). The bases of the modified layer do not contain oxygen. In a thin «white» layer (point 1 in Figure 4), inhomogeneities are seen, which are probably predetermined by a nonuniform oxidation (oxygen content is up to 11.81 %).

A crystalline structure of the modified layer contains discrete elements smaller than 100 nm (Figure 5). In the surface layer at a depth of up to 6 µm, the presence of a crystalline structure is not observed. The modified layer consists of a nanocrystalline alloy having apparently amorphous layers.

In the surface layer of stamped steel, the concentration of the main elements (tungsten, oxygen, carbon), affecting the heat resistance of this layer increased. Tungsten was introduced into the plasma jet in the form of erosion products of the metal electrode (rod), carbon was introduced from the surface in the form of an interlayer, and oxygen is a component of the plasma. The formation of a dense oxidized layer on the surface of the stamped steel closes it from the further oxidation and prevents the contact of metals of the stamped alloy and the die. The distribution of alloying elements across the thickness of the modified layer was determined using an atomic emission spectrometer of a glow discharge. Precision quantitative layer-by-layer analysis of a stamped steel after pulse-plasma treatment with the introduction of tungsten, carbon and oxygen into the plasma showed that alloying elements are located at a depth of up to 80 µm (see Figure 4). In the surface layer, a new material with a high content of alloying elements was formed.

It is expected that additional alloying of a stamped steel surface by carbon and oxygen will reduce the effect of oxygen during operation of the dies. Pulse-plasma treatment of a product and a complex alloying will



Layer depth, µm	C, wt.%	O, wt.%	W, wt.%
5	2.19	11,81	1.28
30	3.83	0	1.20
50	4.36	0	2.14
80	3.50	0	4.36

Figure 4. Results of analysis of alloying with carbon, oxygen and tungsten. Treatment by direct plasma (with a supply of potential)

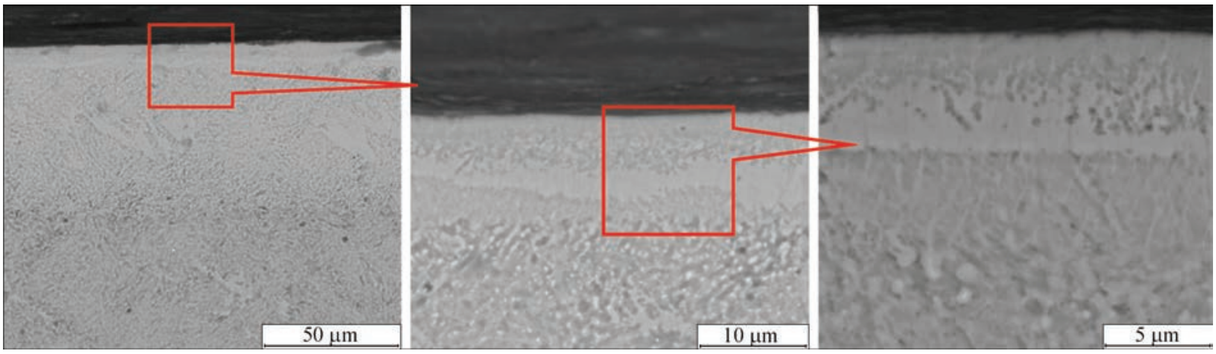


Figure 5. Microscopic analysis of the modified surface layer obtained by supply of a potential. Electron-ion scanning microscope Quanta 200 3D. Etching by Rzeszotarski's reagent

Figure 6. Surface of the die for deep drawing of products from VT-6 titanium sheet: *a* — unmodified; *b* — modified with a pulse plasma by complex alloying with carbon, tungsten and oxygen

increase the service life of the stamping tool surface and eliminate failures because of brittle fractures.

Taking into account the previous work, the working surfaces of a large-sized matrix of the die for hot drawing of a titanium sheet from VT-6 alloy of 3 mm thickness were modified (Figure 6). The working area of the die (matrix) was made of a stamped steel 4Kh4VMFS (DI-22), heat-treated applying a standard technology. The surface of the die was ground after heat treatment $R_z = 1.6$.

Before stamping, a matrix of the die is heated to a temperature of 650–690 °C. At a temperature of 500 °C and higher, on the matrix surface a scale is formed, which significantly deteriorates sliding of the workpiece during shape drawing. The destruction of the die surface, adhesion of titanium on the surface, oxidation and roughness violation are observed (see Figure 6, *a*).

To increase the serviceability of a stamping tool, the surface was modified with a pulse-plasma. Treatment was carried out using a pulse-plasma generator «Impuls» (Figure 1).

Results of industrial tests of a modified pulse plasma stamping tool

Description of the tool	Without mod. t/unit	After mod. t/unit	Efficiency, %
Detachable matrix	24.4	79.35	325
Supporting matrix	4.5	29.23	650
Slotted punch	1.8	7.08	393
Deformation punch	10.85	22.5	207
Forming matrix	18.0	54.0	300

Elastic-plastic deformations of the surface layer in combination with pulsed thermal and electromagnetic influence provide a refinement of the alloy structure and intensify the diffusion mechanisms of alloying elements. Previous studies of reference specimens showed that the modified layer contains such alloying elements as carbon, tungsten and oxygen, has a three-fold increase in hardness and reduced characteristic sizes of crystal structures (< 100 nm). The modified surface has a low roughness.

The dies were used in the technological process of deep drawing of a titanium sheet with a thickness of 3 mm. The sheet was heated to a temperature of 700 °C. After the use of dies on an intended purpose, no traces of oxidation and roughness violation were observed on their surface (see Figure 6, *b*).

Technology and technological equipment for pulse-plasma strengthening of the tool are used in the hardware production of JSC «Cherepovets Steel Rolling Plant». An experience of industrial operation demonstrated that serviceability of the tool made of stamped steel subjected to pulse-plasma treatment, increased by 2–6 times (see Table [10]).

Conclusions

The proposed technology and equipment for pulse-plasma modification provide alloying and nanostructuring of the surface layer of tool steels to a depth of more than 80 μm.

Industrial tests confirmed the effectiveness of modification. After pulse-plasma treatment of working surfaces of a large-sized matrix of the die for hot drawing of a titanium sheet from VT-6 alloy with a thickness of 3 mm, during heating to a temperature of 700 °C the tool of a stamped steel 4Kh5MF1S did not have failures because of adhesion wear, brittle fracture or breakdown. The serviceability of the tool was 2–6 times increased. The only cause for the failure of the tool is incandescence of working surfaces.

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