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The peculiarities of processing of constructional materials by pulse plasma streams

In this paper presented the results of studies of stainless steel samples surface after modification by pulsed plasma flows, processing by single and multiple time. The modification carried out on CPU-30 pulsed plasma accelerator worked at continuously filling mode. The working gas air on pressure 0.04-0,5 mmHg and energy density of plasma 0-40 J/cm^2. The changes of properties of materials investigated by SEM and XRD methods.

The evolution of the grain, as a structural component of steel, depending on the basic processing parameters, was researched. Is shown, that changes in the structure of the steel influence of the microhardness of material surface. The peculiarities of influence of powerful plasma impact on constructional materials is the the formation of hardening nitride and carbide phases.

***Keywords**:* pulse plasma, stainless steel, structure of the steel, constructional materials.

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Конструкциялық материалдарды импульсті плазма ағынымен өңдеу ерекшеліктері

Бұл жұмыста тот баспайтын болат үлгісінің бетіне бірнеше уақыт аралығында импульстік плазмалық ағынмен әсер еткен кезде алынған зерттеу нәтижелері келтірілген. Эксперименттік жұмыс үздіксіз толтыру режимінде жасайтын импульстік плазмалық үдеткіште (ИПҮ-30) жасалды. Жұмыста газ ретінде ауа алынды, қысымы 0,04-0,5 мм сынап бағанасы және плазма энергиясының тығыздығы 0-40 Дж/см^2. Материалдар қасиетінің өзгерісі рентген құрылымдық талдау (РҚТ) және растрлық электрондық микроскопия (РЭМ) әдістерімен зерттелді.

Өңдеу параметрлеріне байланысты болаттың құрылымдық бөлігінің (дәндердің) эволюциясы зерттелді. Болат құрылымының өзгерісі материал бетінің микроқаттылығына әсер ететіндігі көрсетілді. Конструкциялық материалдарға плазма ағынның күшті әсерінің ерекшеліктері – нитрид және карбид фазаларының бекемдеуінің себебі болады.

Түйін сөздер: импульстік плазма, тот баспайтын болат, болаттың құрылымы, конструкциялық материалдар.

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Особенности обработки конструкционных материалов импульсным потоком плазмы

В данной работе представлены результаты исследований поверхности образцов из нержавеющей стали после воздействия импульсными плазменными потоками, обработанных в течении некоторго времени. Модификация осуществлялась на импульсном плазменном ускорителе ИПУ-30, работающем в режиме непрерывного заполнения. В качестве рабочего газа выбран воздух; давление 0,04-0,5мм рт.ст. и плотность энергииплазмы 0-40 Дж/см^2. Изменения свойств материалов исследовались с помощью методов рентгеноструктурного анализа (РСА) и растровой электронной микроскопии (РЭМ).

Была исследована эволюция структурного компонента стали (зерна) в зависимости от основных параметров обработки. Показано, что изменения в структуре стали влияют на микротвердость поверхности материала. Особенности влияния мощного воздействия плазмы на конструкционные материалы являются причиной формирования упрочнения нитрида и карбида фаз.

Ключевые слова: импульсная плазма, нержавеющая сталь, структура стали, конструкционные материалы.

Introduction

Treatment of solid surfaces using pulsed plasma flows is one of the most promising ways to create materials with desired properties, including the nanoscale structure. A feature of this method is the high density power impact on the surface of materials, resulting in melting of the surface layer of the material and the cooling rate of 10^6-10^8 K/s [1-3]. Obviously, the physical parameters of the process leading to the formation of structures of a given size can be achieved only under certain modes of exposure. As shown in our works, pulsed plasma accelerator CPU-30 has a power density of 0.5-5 MW/cm^2 high enough for surface processing, such as semiconductors, metallic materials with melting [4,5]. Well researched work of the accelerator in pulsed mode, when the working gas is injected into the electrode space before applying a high voltage. In the other mode, so called “continuously mode”, at constant initial gas pressure in the chamber, the plasma parameters vary over a wide range. In addition, recently shown that at different gas pressures in the channel of the accelerator can form clots certain form, which may play an important role in the processing of the material.

High heating and cooling speed of the plasma flows promote large temperature gradients on the material, which, in turn, serve as a prerequisite for the activation of chemical reactions on the surface and stimulate the generation and diffusion of radiation-induced defects and etc. In the process of crystallization from the melt in the surface layer of the material may change, leading to the formation of new compounds, the intermediate states are metastable phases. Interest is the study of the mechanism of crystallization in a homogeneous eutectic metal and nonmetal melts, and their relation to structural changes and tribological characteristics.

Experiment

In this paper, the generator of pulsed plasma flows is the accelerator with coaxial electrode geometry CPU-30 [6]. CPU generates a plasma flows with velocities (10-100) · 10^3 m/s and a high density of the ion kinetic energy from 10 eV to 10^3 eV with a pulse duration of ~ 15 µs. Flux density and efficiency of influence depends on the conditions under which the processing is carried out of the target. In the experiment, the target used samples of nickel-chromium alloy 12X18N10T, whose surface has been treated with a residual air pressure in the working channel of the accelerator. Processing in this mode is characterized by a homogeneous energy distribution of the plasma flow on the surface of the study object. Was selected a wide range of operating pressures P = 0,04 ÷ 0,5 mm Hg and energy density Q = 8 ÷ 50 J/cm^2. Treatment was carried out once and multi time. The samples were placed at a distance of 6-10 cm from the end of the central electrode in a plasma focus area.

In this paper, on the basics of 12X18N10T steel samples had studied the mechanism of growth of crystallites of the individual phases at the eutectic solidification of metallic alloys after exposure to plasma. The influence of the main parameters of the plasma treatment on the evolution of grain, and as a result, the surface hardness of stainless steel. In the analysis of experimental data, we used the methods of X-ray diffraction (XRD) and scanning electron microscopy (SEM). Microhardness of the surface was determined by Vickers (HV) method on metallographic microscope “Metaval” under loading of indenter 20g.

Scherer’s method used in the XRD to determine the crystallite size of main phases, it was found that the single effect of the plasma density variation energy Q in a given interval (P = 0.04 mmHg) reduces the size of the main phase of the crystallites were Fe_γ ~ 2 time (Table 1). The greatest effect is achieved by grinding grain Q = 8,5 J/cm^2. The increase of Q causes small fluctuations is shown, while the crystallization process is not stable, then the phase Fe_γ grain size does not change. Such changes in the surface structure can be explained by the conditions of the process, in particular the selected operating pressure at which the CPU-30 to reach a maximum value of the energy density of the plasma flow. The degree of melting in this case may lead to unexpected side effects. For example, the presence of a small amount of ferrite phase in the sample number 1, 3, the amount of which decreases and disappears at the sample number 4.

In addition, the sample number 3, 4, revealed the presence of two phases with the structure Fe_{γ} (austenite) to the parameters of a fcc lattice, close to the parameters of the main phase $Cr_{0,19}Fe_{0,7}Ni_{0,11}$, but differ greatly. The content of the second phase of austenite is approximately one-third of the content of the main phase of γ-Fe. Perhaps this refers to the austenite phase, which is not on the surface, but in the depth of the sample. With Q increasing the second phase of the austenite structure disappears (sample number 5, 6), possibly due to the fact that the process of modifying the sample affects the deeper layers of the sample.

Table 1 – The results of X-ray analysis of stainless steel after treatment

№ обр.	n	Q, J/cm²	a,Å	$2\theta_{max}$, grad.	I_{max}, abs. un.	phase	L, Å
Исх.	0	0	3.5824±0.0006	43.689 2.07018	902	$Cr_{0,19}Fe_{0,7}Ni_{0,11}$	1560
			-	44.554	18.2	Fe_5C_2	-
One time impact							
№1	1	8,5	3.5824±0.0006	43.712	1490	$Cr_{0,19}Fe_{0,7}Ni_{0,11}$	470
			2.8650±0.0007	44.740	56.2	Fe	747
№3	1	19,95	3.5843±0.0006	43.687	622	$Cr_{0,19}Fe_{0,7}Ni_{0,11}$	700
			3.5901±0.0006	43.540	253		
			2.8647±0.001	44.702	27.9	Fe	1135
№4	1	27,1	3.5862±0.0006	43.657	727	$Cr_{0,19}Fe_{0,7}Ni_{0,11}$	560
			3.5949±0.0008	50.737	63.3		
№5	1	34,9	3.5868±0.0006	43.659	751	$Cr_{0,19}Fe_{0,7}Ni_{0,11}$	530
№6	1	39,25	3.5866±0.0006	43.649	815	$Cr_{0,19}Fe_{0,7}Ni_{0,11}$	470
Multi time							
№3	10	27.2	3.5873±0.0006	43.253	455.0	$Cr_{0.19}Fe_{0.7}Ni_{0.11}$	350
			3.6113±0.004			$FeN_{0.056}$	163
№1	20	23.1	3.5896±0.0015	43.326	578.0	$Cr_{0.19}Fe_{0.7}Ni_{0.11}$	480
			3.6104±0.0011			$FeN_{0.056}$	123
№2	30	30.1	3.5892±0.0006	43.325	410.0	$Cr_{0.19}Fe_{0.7}Ni_{0.11}$	440
			3.6162±0.0026			$FeN_{0.056}$	133

With increasing numbers of treatment (up to 10, 20 and 30 time) the structure of the sample surface stainless steel 12X18N10T modified in solid solution in which the main are two phases: the austenite - $Cr_{0.19}Fe_{0.7}Ni_{0.11}$ and iron nitride. The elemental composition of iron nitride is expressed by $FeN_{0.056}$. Small crystallite size may be due to a reduced degree of crystallinity nitride. A small number of samples in the presence of a third phase, which can be identified as Fe_3C - iron carbide.

Effect of plasma treatment parameters Q and P on the crystallite structure apparent when studying the surface of steel 12X18N10T by SEM (Figure 1, 2, 3). To identify the microstructure was chosen mode of electrolytic etching plasma-treated samples for grain.

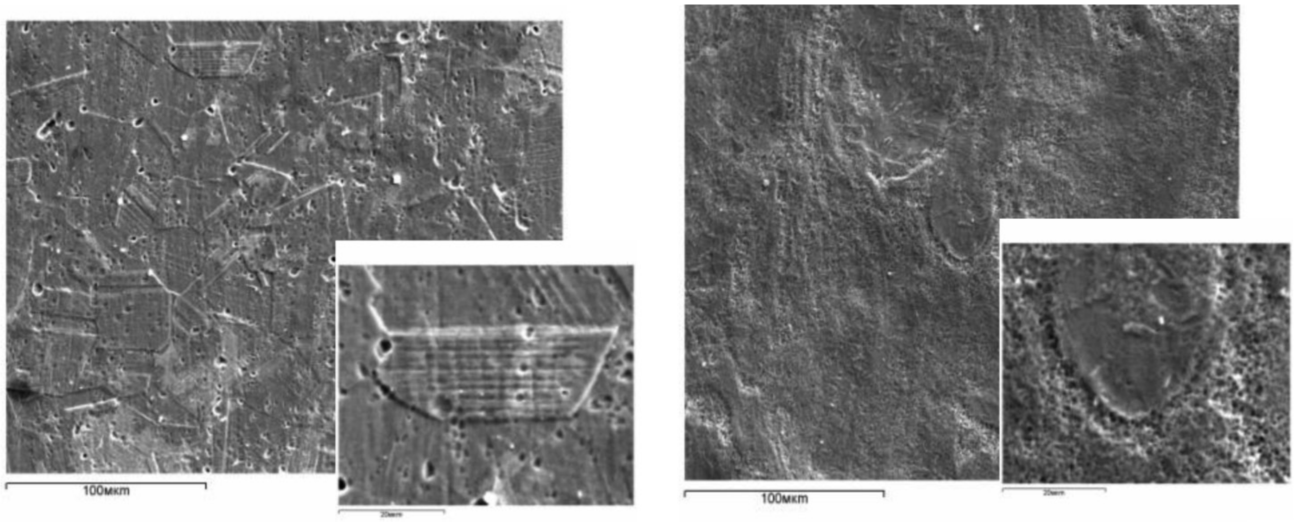

Figure 1 – Topography of the sample number 1 (Q = 8,5 J/cm^2, n = 1), № 4 (Q = 27,1 J/cm^2, n = 1) general view (× 400) and a fragment (× 2000) after treatment with P = 0.04 mm Hg respectively

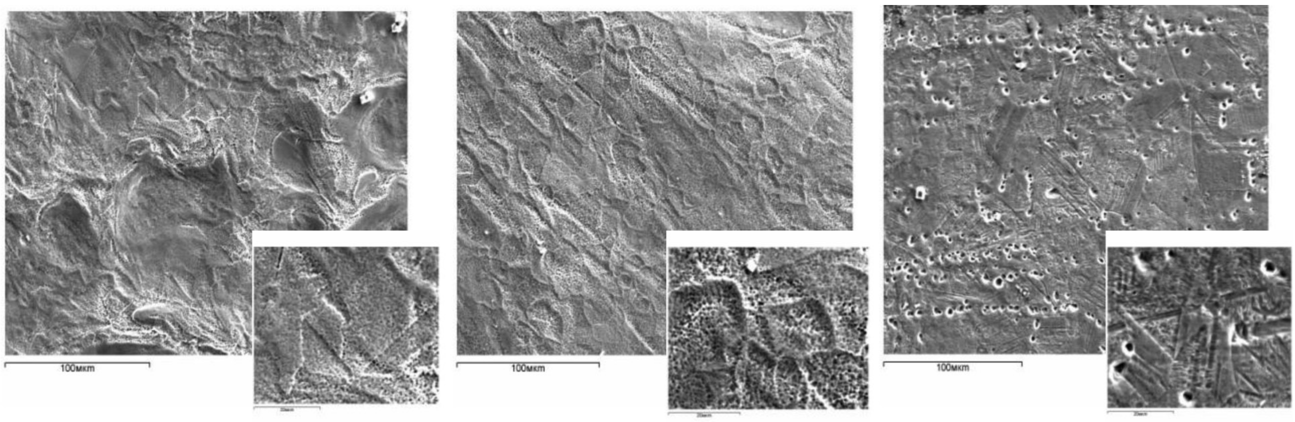

Figure 2 - The surface of the samples № 1 (Q = 4,2 J/cm^2, n = 1), № 3 (Q = 8 J/cm^2, n = 1) and № 4 (Q = 22,8 J/cm^2, n = 1) general view (× 400) and a fragment (× 2000) after treatment at P = 0.1 mm Hg

On the pictures is clearly visible a transformation of grain with increasing of Q and P. The presence of various rack-and-plate-crystalline structure with a distinct defect of twinning on many samples, suggesting the presence of a phase transition of γ-Fe lattice in the α-Fe (martensite phase) with another type of lattice. Since in this case the martensite phase to the austenite is more stable, such a transition should be accompanied by a hardening of the material surface. Fluctuations in grain size, marked XRD associated with the phase transformation, the formation of a well-known compounds and the new, the occurrence of which is not predicted by the equilibrium phase diagram.

As we know, the birth of new compounds proceeds in parallel processes of defect, the result of which is the appearance of micro-inclusions, areas of damage and plastic deformation characteristic of doping impurities and chemically active, directly or indirectly, by affecting the synthesis of new phases and the restructuring of the treated surface. When the concentration of implanted ions stoichiometric values in the matrix formation of compounds that require minimal restructuring. Table. 2 shows the changes in the elemental composition of the samples were 12X18N10T when processed in the given conditions.

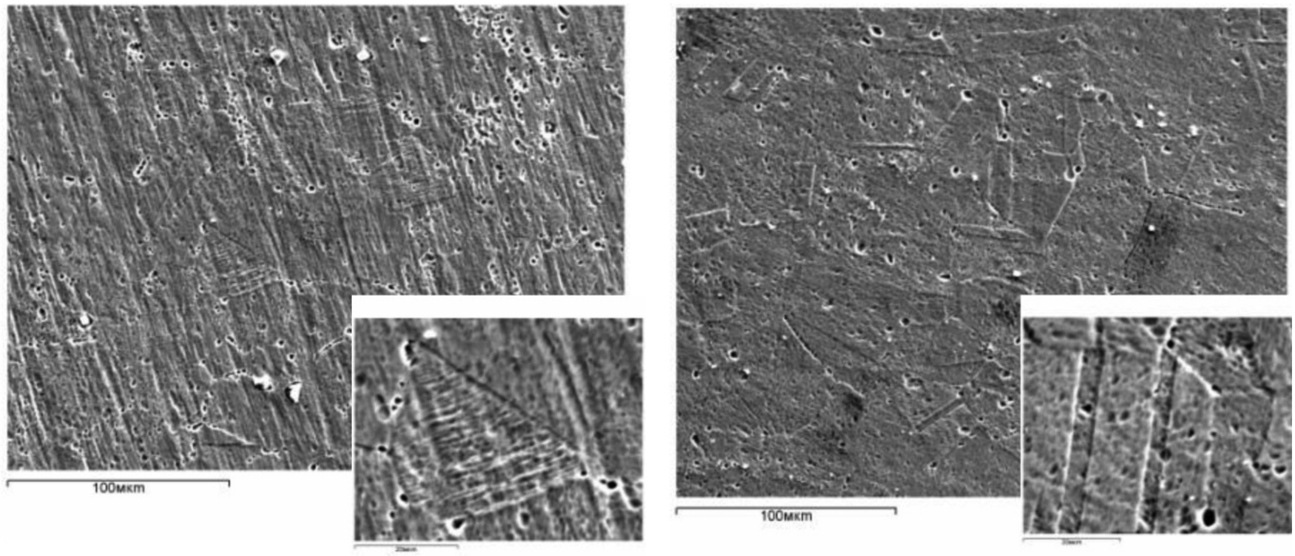

Figure 3 – The surface of the sample number 2 (Q = 10,35 J/cm^2, n = 1), № 3 (Q = 19,9 J/cm^2, n = 1) general view (× 400) and a fragment (× 2000) after treatment with P = 0.5 mm Hg respectively

Table 2 – Stoichiometricchangesin steel12Kh18N10Tafter processing

№ sumple	Al, %	Si, %	Ti, %	Cr, %	Mn, %	Fe, %	Cu, %	Ni, %
4 (initial)	-	0,68	0,21	17,85	1,58	70,61	-	9,07
At air work pressure0,1 mm Hg.								
4 (one time)	-	0,67	0,16	17,96	1,69	70,42	0,39	9,10
3 (10 time)	-	0,67	0,33	17,81	1,40	70,61	0,33	8,85
1 (20 -)	-	0,63	0,26	17,87	1,62	70,32	-	9,30
2 (30 -)	-	0,69	0,24	17,83	1,74	69,78	0,51	9,21
At P=0,5 mm Hg.								
3 (one time)	0,10	0,86	0,20	17,69	1,62	70,31	-	9,23
5 (multi time)	-	0,71	0,21	17,82	1,57	70,31	0,39	8,98

After processing can see the changes of surface microhardness of treated samples, as shown in work [7]. Single exposure leads to an increase of microhardness to 1.5 - 2 times as a result of treatment at a pressure of 0.5 mm Hg, repeated - almost three times at = 0.1 mm Hg.

Conclusion

Studies have shown that repeated treatment is effective. It is shown that the dependence of HV on the multiplicity of effects is almost linear. One of the reasons for the increase in microhardness, apparently, are the structural changes, including the formation of hardening nitride and carbide phases. For example, in work [7] stated that when implanted iron-based alloys with nitrogen ions increase the radiation dose to $4 \cdot 10^{16}$ cm^{-2} at room temperature leads to the appearance of the martensite phase, which when heated to temperatures of 426 K into a tetragonal bcc grid iron nitride. And as in the experiment, surface treatment of steel 12X18N10T done repeatedly, as the plasma gas was used by the air that N_2 is one of the main components, and the results of X-ray analysis showed the presence of nitride phase, we can assume that these conditions impulse excitation can initiate similar phase transformation. Strengthening also helps reduce the size of the crystallites with repeated exposure.

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