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The nanodimension crystallite formation in steel surface after pulsed plasma flow action

Absract. This paper presents the analysis of structural-phase changes in samples of stainless steel AISI 321 (12X18H10T) and AISI 201 (12X15Г9НД), treated by pulsed plasma flows on coaxial plasma accelerator KPU-30. Studied the sequence of phase transitions in modified layers depending on a frequency of treatment. It is shown that after treatment with a small number of shots (n = 2) in the stainless steel samples occurs plasma etching and redistribution of crystallites. At tenfold (n = 10) processing the structure of the surface layer becomes more ordered, appear nanoscale columnar blocks evenly spaced on the surface and their tracks are located mainly at the grain boundaries. The results of SEM and AFM analysis showed the presence of surface melting; this suggests probable structural and phase changes during plasma processing. Changes in the structure of the investigated structural steels, associated with the formation of new phases and the microscopic strains in the crystal lattice, studied by XRD method.

Key words: stainless steel, microstructure, nanoscale inclusions, plasma processing, pulsed plasma accelerator.

Introduction

In modern production, high requirements placed on technological and operational characteristics of construction materials. Currently, the most common among structural materials remain metallic materials. Therefore, the problem of improvement of technological methods of hardening of structural steels, including nanoscale structural modification is relevant.

As it is known, processing of pulsed plasma flows has a number of advantages in comparison with traditional technological processes of thermal-mechanical and chemical-thermal treatments, and methods of exposure based on the use of other types of concentrated energy flows, including laser radiation, high-current electron and ion beams, lasers, etc. [1-4].

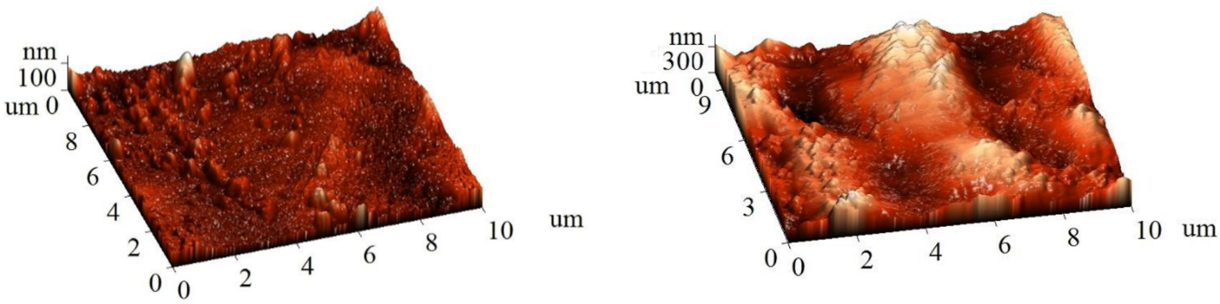
The technologies of surface hardening based on a modifying effect on the metal surface by energy or physical-chemical methods, which radically changes its structure and properties [5-11]. Pulsed plasma treatment is one of the most effective ways of modifying the surface of a solid body.

Earlier in the works [12, 13] have been analyzed changes in the structure of steel and stainless steel were processed on the accelerator KPU-30. In ordinary carbon steels observed strong reduction of the crystallites size with increasing energy, but in stainless steels this is not happening. Furthermore, the iron nitride when exposed to nitrogen plasma uniquely identified only in stainless steel. Thus, to find out the reason of hardening materials, it is necessary to conduct research on materials that combine the properties of carbon and stainless steel and manufactured with the same technology. Stainless steel AISI 201, in which expensive nickel to stabilize the austenitic structure partially replaced by manganese and nitrogen, has long established itself as an effective substitute for the standard chromium-nickel steels. This paper presents the analysis of structural-phase changes in the structural steel samples processed by pulsed plasma streams of stainless steels AISI 201 and AISI 321.

Experiment details

Samples of the test material were exposed to pulse treatment on coaxial plasma accelerator KPU-30 with a residual air pressure of 13,3 Pa. At a voltage of 20-22 kV, the energy density of the plasma flow varied in the range of 14,2 ÷ 15,4 J/cm^2. During the experiment, the samples were placed in the working chamber at a distance of 7 cm from the end of the center electrode in the area of plasma focus.

Using atomic force microscopy (AFM) it is possible to descry in detail the topography of the surface in two forms images on the plane and in 3D format. Thus, obtained spatial images of three different areas. Figure 1 shows the AFM images of the surface of steel samples AISI 201 and AISI 321, exposed the twofold influence of plasma flow.



a b

Figure 1 – The AFM images of the surface of steel samples AISI 201(a) and AISI 321 (b)

As shows data analysis of steel AISI 201, the melting of the surface at twofold action leads to the local formation of blisters, also there are areas with columnar crystallites, which formed in a perpendicular direction to the surface (Fig. 1a). Tenfold treatment leads to an increased efficiency of the double treatment, and the columnar crystallites moved predominantly to the grain boundaries (blocks). The paper presents quantitative estimates of the size of crystallites. The AFM analysis of processing results of steel AISI 321 showed that, in contrast to steel AISI 201, the height of the columnar crystals are more than for steel AISI 201, already at twofold treatment (Fig. 1b), but, as well as in case of AISI 201 (n = 10), the columnar crystallites are mainly located at the grain boundaries. In some parts of the steel AISI 321 and as well as in the steel AISI 201 traces of the formation of blisters. Furthermore, for this grade of steel samples were not detected traces of delamination surface that, apparently, it is not typical for this steel. According to the preliminary results, we can conclude that under certain parameters of the plasma exposure, the modification of the surface structure of structural steel by fusion, accompanied by destruction of the crystal bonds and plasma etching.

To determine the changes in physical and mechanical properties presented studies of the microhardness on metallographic microscope "METAVAL" by Vickers method. Measurements of surface microhardness of the steels AISI 201 and AISI 321 processed by pulsed plasma streams carried out on all three areas with different surface topography.

Experimental data analysis

Comparing the results of measuring the microhardness in different areas of the twice treated surface with the original, it was found that surface hardening is uneven, with the presence of local areas with both elevated and reduced hardness. Comparing these areas with the results of the AFM analysis, in the first case, the decrease of hardness corresponds to "delamination" of the surface (1st area), in the second case - blister formation (2nd area), and in the third case, the increase of microhardness can be associated with a "leveling" of the surface and streamlining of placing the columnar crystallites over the entire surface area (3^d area). As a result of the tenfold processing, as shown by measurements, the surface hardening at all three areas increases slightly (~ 100 MPa), thus there are areas where the microhardness is very unstable (3^d area). Comparing with the results of the AFM analysis, we can assume that this may be due to the redistribution of columnar crystallites (a preferential distribution at the grain boundaries) and increase their size (height). Microhardness measurements of the steel AISI 321, performed after double treatment, showed its reduction from baseline to a significantly greater extent than for steel AISI 201. The results of tenfold treatment, on the contrary, show that typical growth of microhardness is more significant for AISI 321 (to ~ 150 ÷ 300 MPa) than in the previous grades of steel, but the average microhardness is much less than earlier obtained data [7,9]. Perhaps, this is due to the formation of block structure, typical for plasma etching.

Data analysis by average values of surface microhardness of both grades examined steels showed that, on average, a greater growth of microhardness is typical for AISI 321 (n=10). Despite the fact, that changes in microhardness insignificant, however, they take place, moreover, the results of SEM and AFM showed the presence of surface melting, this suggests probable structural and phase changes during plasma processing.

Data for pulse plasma treatment of stainless steel grades AISI 201 and AISI 321 are given in table 1. In the initial state the samples of carbon steel were monophasic, but in this case, monophasic ferrite with lattice parameter a = 2,8686 ± 0,0007 Å (according to the standard diffractometric data parameter of iron equal to a = 2,8664 Å) (table 1).

Table 1 – The data of RSA tenfold processed steel grades AISI 201 and AISI 321

Sample status	n	Phase		a, Å	L, Å
Data for AISI 201 (12Х15Г9НД)					
Untreated	0	monophase	(Fe,C) austenite	3,6057 ± 0,0006	–
Treated	10	multiphase	(Fe,C) austenite	3,5958 ± 0,0006	1900
			($FeN_{0,076}$) iron nitride	3,6263 ± 0,0007	270
Data for AISI 321 (12Х18Н10Т)					
Untreated	0	monophase	(Fe,C) austenite	0,35824 ± 0,00006	156,0
Treated	10	multiphase	(Fe,C) austenite	0,35873 ± 0,00006	35,0
			($FeN_{5,6}$) iron nitride	0,36113 ± 0,0004	16,3

Sample steel AISI 201 has two phases. One of these phases belongs to austenite with the lattice parameter a = 3,5958 ± 0,0006 Å. The parameter of austenite is somewhat less than the initial sample, which may be associated with distortion of the crystal lattice of steel during plasma treatment. The second phase belongs to iron nitride $FeN_{0,076}$ with the lattice parameter equal to a = 3,6263 ± 0,0007 Å. On a comparison between intensities of diffraction lines of iron nitride and austenite for the same planes, we can conclude that the nitride is not the dominant phase. It is possible that the iron nitride is in the surface layer, and the austenite is a little deeper. In this case, the thickness of the nitride is small. Therefore, the data X-ray structural analysis confirmed the possibility of increasing of the microhardness after processing the surface of a material with a pulsed plasma. Thus, the surface hardening can be connected with a formation in the investigated steel new phase $FeN_{0,076}$ as was assumed in earlier works [7-9].

As a result of tenfold processing steel AISI 321 by plasma flows at a pressure of 13,3 Pa detected new phases – iron nitride $FeN_{5,6}$,and possibly iron carbide Fe_3C. The number of lines Fe_3C is extremely small for identification. As you can see, there is a broadening of the lines belonging to the iron nitride, in comparison with the lines of the austenite. The broadening of the lines of iron nitride is associated with distortion of nitride lattice, the degree of distortion, which increases with increasing frequency of treatment. According to the X-ray analysis, shown in table 1, the crystallite size of the austenite is reduced more than in 4 times with frequency of treatment n=10 in comparison with the untreated AISI 321. In addition, after ten times of treatment, the size of crystallites of both phases are identical. Therefore, multiple pulse plasma processing the most effective for the grinding of the austenite crystallites and, in particular, of iron nitride as in the case of steel AISI 201.

Conclusion

According to the results of work, we can conclude that the treatment with pulsed plasma flows leads to a change of physical and mechanical properties owing to the structural-phase changes and defect formation, which confirmed by the following analysis methods. The results of SEM analysis revealed that after treatment plasma etching takes place, and a redistribution of the crystallites. With increasing the frequency of treatment by pulses of plasma (n=10) the etching pattern becomes more intense, and for steel AISI 321 has been identified already at double treatment in comparison with steel AISI 201.

The AFM analysis revealed that the surface of test material at twofold treatment detected traces of blister formation, the presence of layered structure and tracks, the formation of columnar structures, which may be due to the planar and linear defects. At tenfold processing, the structure is more ordered, columnar blocks arranged relatively uniformly over the surface and their tracks are located mainly at the grain boundaries.

The results of metallography, we can conclude that with multiple treatments (n=10) samples, the surface hardness is increased, and for the second type of steel (AISI 321), the effect is more pronounced than for the other (AISI 201), which is consistent with the results of the SEM analysis.

All the above is confirmed by X-ray diffraction analysis, which revealed changes in structure of the investigated steels, is related to the formation of a new phase of iron nitride and the microscopic strains in the crystal lattice that may be responsible for hardening.

In general, by the results of performed work, it concluded that treatment with pulsed plasma flows leads to a change of physical and mechanical properties at the nanoscale, and this could be due to structural phase changes and defect formation. Furthermore, the results of SEM analysis revealed that after treatment plasma etching, the formation and redistribution of nanosized crystallites take place.

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