The stainless steel structure after pulsed plasma flow interaction

A.M. Zhukeshov*, A.T. Gabdullyna,
M. Mukhamedryskyzy and A.U. Amrenova

Physics and Technology Department,
Al-Farabi Kazakh National University,
Al-Farabi ave. 71, Almaty, Kazakhstan
Fax: +7 (7273) 77-34-01
Email: azhukeshov@gmail.com
Email: gabdullyna71@gmail.com
Email: mukhamedryskyzym@gmail.com
Email: amrenova.asem77@gmail.com
*Corresponding author

Abstract: The paper presents the results of studies of modification of the surface structure of structural steels AISI 321 (12X18H10T) and AISI 201 (12X15Г9НД) after plasma treatment with several pulses. The topology of the surface and the evolution of the relief are studied depending on the main processing parameters. In particular, the sequence of phase transitions in modified layers depending on the processing multiplicity was studied. It is shown that under a small number of treatments ($n = 2$) in investigated stainless steel samples may occur plasma etching and redistribution of crystallites. In addition, in samples detected traces of blister formation and the presence of a layered structure, which can be due to planar and linear defects. When increasing the multiplicity of treatments to 10, the structure becomes more ordered, the columnar blocks are located relatively evenly over the surface and their tracks are located mainly along the grain boundaries. The changes in the structure of the studied structural steels are associated with the formation of new phases (iron nitride) and microarrays in the crystal lattice. A decrease in crystallites size to 16 nm was detected. It is shown that the multiple pulse plasma processing is most effective for grinding austenite crystallites, especially the iron nitride in stainless steel.

Keywords: plasma processing; stainless steel; microstructure; pulsed plasma accelerator; surface modification.

Reference to this paper should be made as follows: Zhukeshov, A.M., Gabdullyna, A.T., Mukhamedryskyzy, M. and Amrenova, A.U. (2019) 'The stainless steel structure after pulsed plasma flow interaction', *Int. J. Nanotechnol.*, Vol. 16, Nos. 1/2/3, pp.196–203.

Biographical notes: A.M. Zhukeshov entered the Faculty of Physics of KazSU named after Kirov, from where he was transferred to the Faculty of Physics of the St. Petersburg State University and graduated in 1994. A working career in Al-Farabi KazNU began in 1998 at the Department of Optics and Plasma Physics as a specialist of the 1st category, since 2000 he started teaching as an assistant. In 2003, he defended his thesis on the specialty "01.04.07-Physics of Condensed Matter". In 2009, he received the title of Associate Professor. In 2010, under the leadership of Academician F.B. Baimbetov defended his Doctoral thesis on the specialty 'Plasma Physics'. Currently, he works as a

Professor at the Department of Plasma Physics and Computer Physics. Scientific interests are related to the development, and application of plasma accelerators, nanotechnology, and industrial electronics. He has more than 200 scientific publications, published two monographs, a number of textbooks and manuals. He is the head of scientific projects financed by the Ministry of Education and Science of the Republic of Kazakhstan.

A.T. Gabdullyna, Acting Associate Professor, candidate of Physical and Mathematical Sciences. In 1988, she entered the Kazakh State University named after Kirov to the Faculty of Physics. Since 1995, she works in KazNU named after al-Farabi. She passed all stages of professional growth from a laboratory assistant to a university teacher. In 2010, she defended her thesis on the specialty "01.04.07-Physics of Condensed Matter". Since 1995, she has been actively involved in the research work of the Laboratory of Pulsed Plasma Accelerator of the Department of Plasma Physics. She is a leading researcher in the laboratory. She has more than 80 publications. Since 2012, she has been managed scientific projects. Scientific directions of activity: interaction of plasma with materials, vacuum and plasma technology, diagnostics of plasma, modelling of plasma processes.

M. Mukhamedryskyzy is a PhD student of Al-Farabi Kazakh National University. In 2010, she graduated from Al-Farabi KazNU. From the same university she received her Master's degree in the specialty "6M060400 – Physics" in 2013. During the training in the magistracy, she passed an internship at the University of Cadiz (Cadiz, Spain). In 2012, she is training at the Belgorod State National Research University (Belgorod, Russia) by academic mobility. She takes part in research work in the Laboratory of Pulsed Plasma Accelerator. Has 12 publications.

A.U. Amrenova, Acting Associate Professor, candidate of Physical and Mathematical Sciences. Scientific activity has been engaged since 2001. Among the scientific achievements: important results on the dynamics of the formation of plasma clusters in a pulsed plasma accelerator, as well as the development of new diagnostic techniques. She has more than 80 published works. In 2007, she defended her thesis on the specialty "01.04.08-Physics of Plasma". In 2012, she became the head of the scientific project. She is a senior researcher at the Laboratory of Pulsed Plasma Accelerator of the Department of Plasma Physics. The range of scientific interests: plasma diagnostics, development, and application of plasma accelerators, vacuum technology.

This paper is a revised and expanded version of a paper entitled 'The structure of stainless steel after influence of pulsed plasma streams' presented at *IX Conference NOR*, Russia, 5 April, 2018.

1 Introduction

Currently, the problem of improving the technological methods of hardening of metal materials, including nanoscale structural modification by concentrated energy flows, is an urgent task in view of the complexity and diversity of the processes taking place.

In this aspect, pulsed plasma treatment is one of the most effective ways of modifying the surface of a solid body [1–5]. In particular, treatment with high-temperature plasma flows leads to hardening of the surface and increase of wear resistance of industrial steels. During rapid heating and melting of the treated surface, significant temperature gradients that occur in the surface layer of the material under the pulsed plasma interaction, contribute to a high rate of diffusion of plasma ions deep into the modified layer. Rapid cooling of the liquid phase leads to structural changes in the surface layer, the formation of fine-grained and quasi-amorphous structure in the following repeated melts [6,7]. Plasma also serves as a source of alloying elements, which are embedded in the modified layer.

High heating and cooling rates of 10^6–10^8 K/s under the pulsed action of plasma flows (PPF) contribute to the creation of large temperature gradients on the material surface, which, in turn, serve as a prerequisite for activating chemical reactions on the surface, stimulate the generation and diffusion of radiation defects, etc. In the process of crystallisation of the melt, in the surface layer of the material changes are possible, leading to the formation of new compounds, intermediate states, metastable phases.

The presented results are included in the cycle of works carried out by the authors on the study of the impact of PPF on structural steels [8,9]. It was shown an increase in hardness in steels, but a detailed picture of surface modification remained inaccessible. In this regard, it is currently of interest to study the details of microstructure by AFM under the influence of pulsed plasma flow and determine the size of the structure, as well as the possibility of obtaining nanomaterials under such influence.

2 Experiment

In the present work, the generator of pulsed plasma flows is an accelerator with the coaxial geometry of electrodes KPU-30 (coaxial plasma accelerator) [4,5]. The KPU forms plasma streams with velocities $(10\div100) \times 10^3$ m/s and high kinetic energy density of ions from ~100 eV to 10 keV with pulse duration ~7 μs. The flux density and effectiveness of the action depend on the conditions under which the target is treated. As the materials for the study were chosen stainless structural steels grades: 12Х18Н10Т or its American analogue – AISI 321 (the American Iron and Steel Institute) and 12Х15Г9НД, respectively AISI 201.

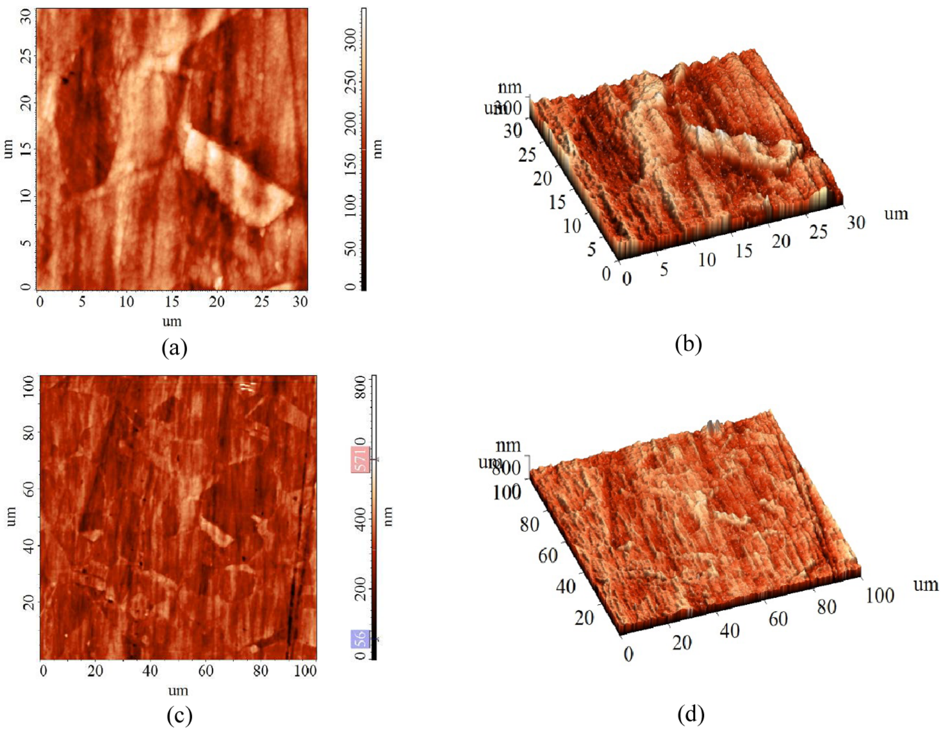
Table 1 Processing parameters of stainless steel samples of AISI 201 and AISI 321

Parameters	*Values*
Gas	Air
Residual gas pressure	13.3 Pa
Charging voltage	20÷22 kV
Energy flux density	14.2÷15.4 J/cm^2
Pulse duration	10÷20 μs
Multiplicity of pulses	2, 10

The samples of the studied material were exposed to pulsed treatment on a coaxial plasma accelerator KPU-30 at a residual air pressure of 13.3 Pa at the processing parameters given in Table 1. As can be seen from the table, at a voltage of 20÷22 kV, the energy density of the plasma flows varied in the range of 14.2÷15.4 J/cm^2. During the experiment, the samples were placed in a working chamber at a distance of 7 cm from the end of the central electrode in the plasma focus zone. Preliminary samples were mechanically polished and chemically polished in an acid environment.

Using atomic force microscopy (AFM, NTegra) 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. Figures 1 and 2 show the AFM images of the surface of all samples under study: before processing and exposed to twofold and ten-fold influence of plasma flows.

Figure 1 The AFM images of the surface of initial steel sample AISI 201 (12Х15Г9НД): (a), (c) – sample profile (30 × 30 μm); (b), (d) – (100 × 100 μm) (see online version for colours)



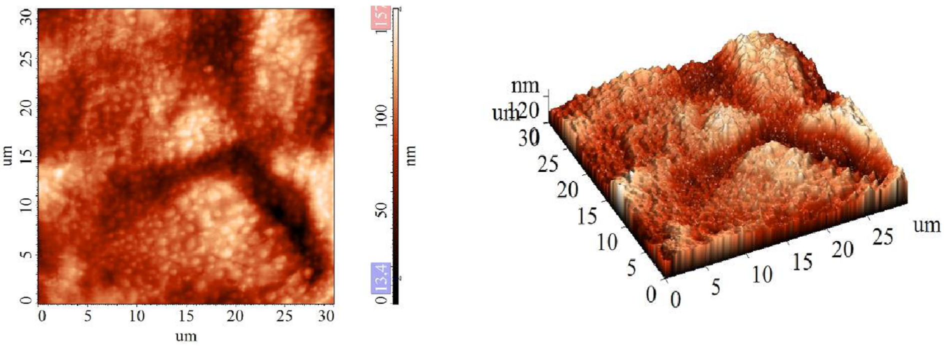
As shown by AFM analysis (Figure 1) on the initial surface is clearly seen etched on the grain structure, obtained by electrolytic etching at the stage of sample preparation of test samples.

Block structure becomes more distinct after plasma exposure, which confirms the possibility of plasma etching at the nanoscale with the specified parameters of treatment even without preliminary etching of the initial surface (Figure 2).

In some cases, plasma etching is accompanied by a certain 'delamination' of the surface structure and clearly defined contours of the track lines. Their contours are clearly visible at large magnifications (Figure 3).

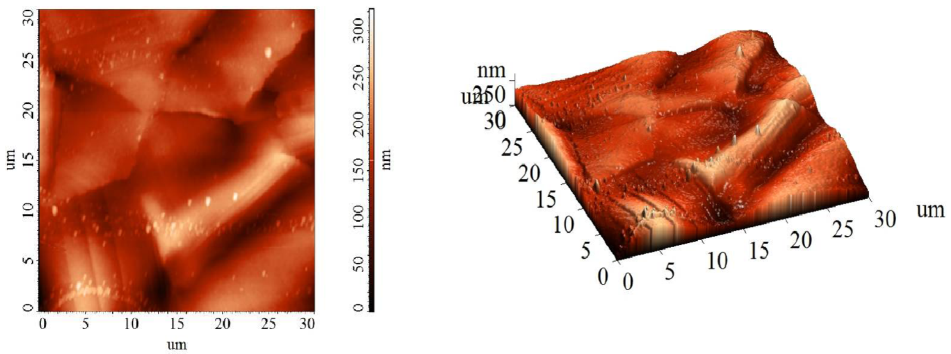
In addition, when twofold treatment surface melting leads to the local formation of blisters and even the opening of blister plaques (Figure 4(a)), and there are areas with columnar crystallites formed in the direction perpendicular to the surface (Figure 4(b)).

Figure 2 Surface relief of the sample steel AISI 201 after twofold processing without pre-etching: (a), (b) – sample profile No. 4 at 30 × 30 μm and 100 × 100 μm (2nd area, P = 13.3 Pa, Q = 12.5 × 10^4 J/m^2, U = 19 kV) (see online version for colours)



(a) (b)

Figure 3 Laminated structure of twofold-treated steel AISI 201 (sample No. 2), obtained by AFM: (a), (b) – sample profile at 30 × 30 μm and 100 × 100 μm (1st area of sample No. 2) (see online version for colours)



(a) (b)

Ten-fold treatment leads to the strengthening of the effect of twofold treatment, and columnar crystallites move mainly to the boundaries of grains (blocks) in accordance with Figure 5. The paper presents quantitative estimates of crystallite sizes.

The AFM analysis of processing results of steel AISI 321(12X18H10T) showed that, in contrast to steel AISI 201 (12X15Г9НД), the height of columnar crystals already at twofold processing is much more than is typical for AISI 201, but, as well as in case of AISI 201 (n = 10), the columnar crystallites are mainly located at the grain boundaries. In some areas of the steel AISI 321, as well as for the steel AISI 201 it is noticeable traces of blistering formation. Furthermore, for this grade of steel it is not detected traces of delamination surface that, apparently, it is not typical for this steel.

Changes in the structure of the investigated structural steels, associated with the formation of new phases and microarrays in the crystal lattice, were investigated by X-ray diffraction analysis on diffractometer D8 ADVANCE (Bruker AXS, Germany).

Based on the X-ray data, the AISI 201 steels have two phases: austenite with a lattice parameter a = 3.5958 ± 0.0006 Å (austenite parameter is somewhat less than the initial sample, which may be associated with distortion of the crystal lattice of steel during plasma treatment.), and iron nitride $FeN_{0,076}$ with a lattice parameter equal to $a = 3.6263 \pm 0.0007$ 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.

Figure 4 Surface relief of sample No. 2 (2nd area) ($P = 13.3$ Pa, $Q = 14.15 \times 10^4$ J/cm^2, $U = 21$ kV, $n = 2$) (see online version for colours)

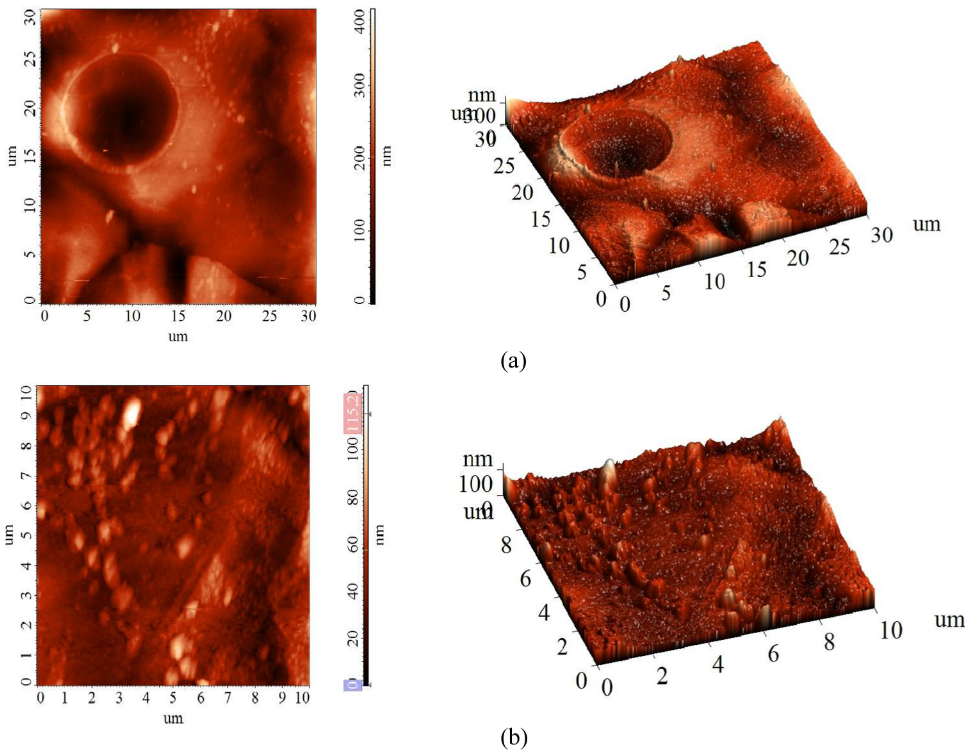
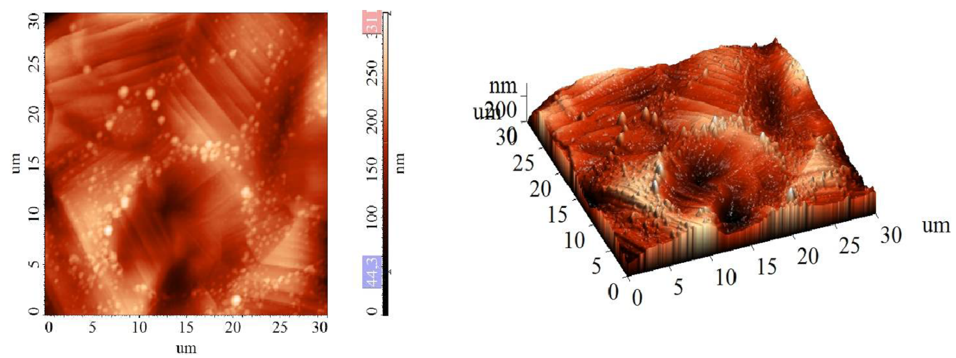


Figure 5 AFM images of AISI 201 steel surface after ten-fold treatment ($P = 13.3$ Pa, $U = 20$ kV, $Q = 15.7 \times 10^4$ J/cm^2) (see online version for colours)



Ten-fold treatment with plasma flows of AISI 321 steel at a pressure of 13.3 Pa is also accompanied by the formation of new phases: $FeN_{5,6}$ iron nitride and Fe_3C iron carbide, although the number of Fe_3C lines is extremely small for identification. According to the X-ray analysis, the austenite crystallites size is reduced more than in four times on multiplicity of treatment $n = 10$ in comparison with the untreated AISI 321. In addition, after ten-fold treatment, the size of crystallites of both phases are identical. Therefore, multiple pulse plasma treatment is more effective for the grinding of the austenite crystallites and, in particular, of iron nitride as in the case of AISI 201 steel.

3 Conclusions

AFM analysis on the surface of the material under study at twofold treatment detected traces of blister formation, the presence of a layered structure and tracks, and the formation of columnar structures, which can be caused by planar and linear defects. At ten-fold processing, the structure is more ordered, the columnar blocks are located relatively evenly over the surface and their tracks are located mainly along the grain boundaries.

According to the research results, it can be concluded that changes in the structure of the investigated steels are associated with the formation of a new phase – iron nitride and a reduction in the size of crystallites to 16 nm, and this can lead to hardening of the material [10–13]. It is shown that the multiple pulse plasma processing is the most effective for the grinding austenite crystallites, especially, the iron nitride in stainless steel.

Acknowledgement

This work was supported by project of MES RK No. AP05130108.

References

1 Zhukeshov, A.M., Baimbetov, F.B. and Ibraev, B.M. (2009) *Impulsnyye uskoriteli plazmy i ikh tekhnologicheskoye primeneniye*, Kazak universiteti, Almaty.

2 Garkusha, I.E., Byrka, O.V., Chebotarev, V.V. Derepovski, N.T., Müller, G., Schumacher, G., Poltavtsev, N.S. and Tereshin, V.I. (2000) 'Properties of modified surface layers of industrial steels samples processed by pulsed plasma streams', *Vacuum*, Vol. 58, Nos. 2–3, pp.195–201.

3 Tereshin, V.I., Bandura, A.N. and Bovda, A.M. (2002) 'Pulsed plasma accelerators of different gas ions for surface modification', *Rev. Sci. Instrum.*, Vol. 73, p.831.

4 Cherenda, N.N., Uglov, V.V., Anishchik, V.M., Stalmashonak, A.K., Astashinski, V.M., Kuzmitski, A.M., Thorwarth, G. and Stritzker, B. (2007) 'Modification of AISI M2 steel tribological properties by means of plasma mixing', *Vacuum*, Vol. 81, No. 10, pp.1337–1340.

5 Tereshin, V.I. Bandura, A.N., Byrka, O.V., Chebotaev, V.V., Garkusha, I.E., Shvets, O. and Taran, V. (2004) 'Coating deposition and surface modification under combined plasma processing', *Vacuum*, Vol. 73, Nos. 3–4, pp.555–560.

6 Chebotarev, V.V., Garkusha, I.E., Tereshin, V.I. and Derepovski, N.T. (1999) 'Surface structure changes induced by pulsed plasma streams processing', *Problems of Atomic Science and Technology, Plasma Physics*, Vol. 3, pp.273–275.

7 Gasin, D.A., Zhusupkeldiev, Sh, Matvienko, V.V. and Uryukov, B.A. (1990) 'Modifikatsiya metallicheskoy poverkhnosti impul'snym potokom plazmy' [Modification of metal surface by pulsed plasma flows]', *Vzaimodeystviye izlucheniya plazmennykh i elektronnykh potokov s veshchestvom [Interaction of Plasma and Electron Streams with Matter]*, Frunze, p.121.

8 Zhukeshov, A.M. and Gabdullina, A.T. (2007) 'Izmeneniye razmera zerna i tverdosti konstruktsionnykh materialov posle impul'snoy plazmennoy obrabotki' [Changes in grain size and hardness of structural materials after pulsed plasma treatment]', *Problemy evolyutsii otkrytykh system [Problems of Evolution of Open Systems]*, Vol. 2, No. 9, pp.40–45.

9 Zhukeshov, A.M., Gabdullina, A.T., Pak, S.P., Amrenova, A.U., Mukhamedryskyzy, M. and Mombayeva, A. (2013) 'Vliyaniye impul'snykh plazmennykh materialov na fiziko-mekhanicheskiye kharakteristiki poverkhnosti nerzhveyushchey stali' [Effect of pulsed plasma flows on the physical and mechanical properties of the stainless steel surface]', *Conference materials: Aktual'nyye problemy sovremennoy fiziki [Actual Problems of Modern Physics]*, Almaty, Kazakhstan, pp.154–155.

10 Zhukeshov, A.M., Gabdullina, A.T., Amrenova, A.U., Pak, S.P., Mukhamedryskyzy, M. and Moldabekov, Zh.M. (2013) 'K vozdeystviyu impul'snoy plazmy na poverkhnost' nerzhaveyushchey stali' [For a pulsed plasma influence on surface of stainless steel]', *Izvestiya NAN RK, seriya fiziko-matematicheskaya [News of the National Academy of Sciences of the RK, Series: Physics and Mathematics]*, Vol. 2, No. 288, pp.71–74.

11 Zhukeshov A.M. (2007) 'Plasma diagnostics in a pulsed accelerator used for material processing', *J. Phys: Conf. Ser.*, Vol. 63, p.012014.

12 Zhukeshov, A.M. and Gabdullina, A.T. (2007) 'Vliyaniye parametrov impul'snoy plazmennoy obrabotki na tribologicheskiye kharakteristiki nerzhaveyushchey stali' [Influence of pulsed plasma treatment parameters on tribological properties of stainless steel]', *Vestnik NYATS RK. [Bulletin of NNC RK]*, Vol. 2, No. 30, pp.28–31.

13 Zhukeshov, A.M., Gabdullina, A.T., Amrenova, A.U., Batani, D., Mukhamedryskyzy M. and Moldabekov, Zh.M. (2017) 'Struktura nerzhaveyushei stali posle vozdeistviya impulsnyh plazmennyh potokov' [The stainless steel structure after pulsed plasma flow interaction]', *3d Conf. Mat.: Fizika i tehnologiya nanomaterialov i struktur [Physics and Technology of Nanomaterials and Structures]*, Kursk, Russia, pp.59–65.