Journal of Nano- and Electronic Physics
Vol. **6** No 3, 03066(3pp) (2014)

Журнал нано- та електронної фізики
Том **6** № 3, 03066(3cc) (2014)

Hardening of Structural Steel by Pulsed Plasma Treatment

A.M. Zhukeshov*, A.T. Gabdullina†, A.U. Amrenova, S.A. Ibraimova

Open-type National Nanotechnology Laboratory, Al-Farabi Kazakh National University, Almaty, Kazakhstan

(Received 19 May 2014; revised manuscript received 07 July 2014; published online 15 July 2014)

Here the results of modification of structure of materials at pulsed plasma processing on CPA-30 accelerator are presented. The changes of physical-mechanical properties by reason of structure and phase transformation in material at difference plasma parameters impact are observed.

Keywords: Plasma, Accelerator, Structure, Crystalline, New phase, Microhardness.

PACS numbers: 41.75. – i, 61.80. – x, 81.15. – z, 81.40. – z

Pulsed plasma accelerators are traditionally used for surface treatment of metals in order to modify physical and mechanical properties of their surfaces. In our research we used a pulsed plasma accelerator CPU-30 with a coaxial electrode system. An important advantage of pulsed accelerators is possibility to vary plasma parameters over a wide range. The accelerator generated a plasma flow with a wide energy range of ions 0.02-10 keV.

The exposure duration was about 20 microseconds, the concentration of particles in the plasma stream varied from 10^{12} to 10^{15} cm^{-3}. At a discharge current of 100-400 kA, the energy density of the plasma flow incident on the sample was 10-100 J/cm^2.

The experimental results showed that the main parameter affecting changes in the surface properties of the above materials was the energy density of the plasma flow. When the energy density was as high as 50 J/cm^2, plasma treatment caused melting of the surface to a depth of several tens of microns. Subsequent recrystallization of the molten layer altered the structure of the layer with formation of microcrystalline structure. The structure was modified under the action of several interrelated factors, the main of which was high speed of cooling of the molten layer, a shock wave and diffusion of embedded particles.

The structure of layers modified by pulsed plasma was studied using scanning electron microscopy (Quanta 200i 3D), X-ray diffraction analysis (D8 ADVANCE Bruker AXS), atomic force microscopy (AFM) (Integra Terma) and measurements of microhardness (METAVAL).

For plasma treatment, the plasma formed in a discharge in air was used. In the experiment, various grades of steel were used as test samples. It was found that in case of melting, on the surface polycrystalline layers were formed, the structure of which depended on the energy density. The average crystallite size ranged from 20 nm to 150 nm, with maximum values corresponding to high energy.

It was also determined that the surface morphology of various samples depended on the number of treatments. For example, two-pulse ($n = 2$) plasma processing of steel grades 12H15G9ND and 12H18N10T caused fragmentation of the surface structure and formation of polygonal blocks of various dimensions differing by orders of magnitude.

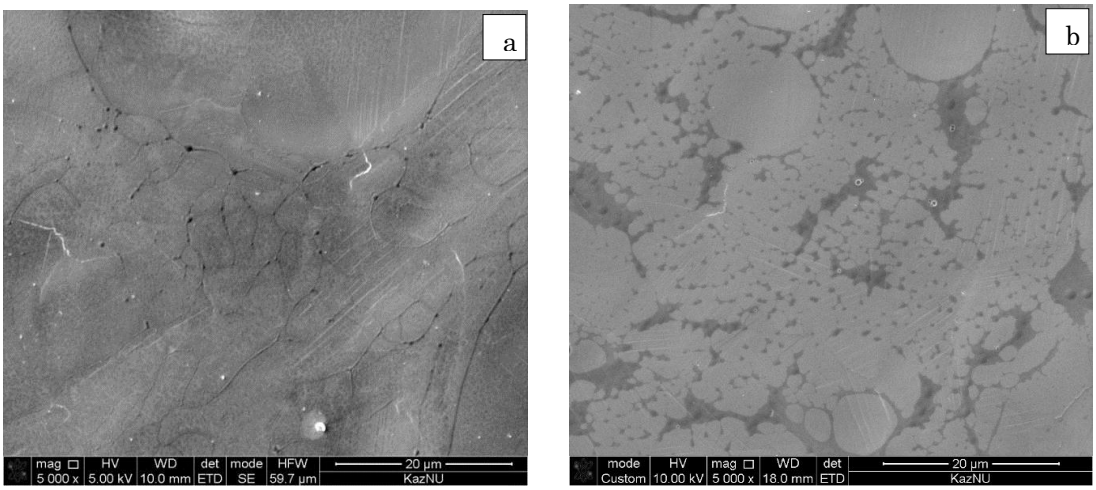

Fig. 1 – AFM image of the 12H15G9ND steel surface after tenfold treatment ($P = 13.3$ Pa, $U = 20$ kV, $Q = 15.7 \times 10^4$ J/m^2)

* zhukeshov@physics.kz
† a_gabdullina@mail.ru

The article was reported at the International Conference «The Advanced Technology, Equipment and Analytical Systems for Materials», Kursk, 13-14 May, 2014

As shown in Figure 1a after double pulsed plasma surface treatment steel 12H15G9ND observed predominance of grains elongated pentagonal and rectangular shape with coarse and fine structure, indicating the formation of local sites with possible changes in the physical and mechanical characteristics. At this magnification clearly visible traces of tracks reminiscent of series-connected small depressions that indicates the formation of not only volume, but also linear defects. With increasing multiplicity of pulses to 10 (Figure 1b), surface melting occurs with the "bulk" of convex structures, which are clearly visible when the image is enlarged to 5000 times. Convex education have finished contour and vary greatly in size and resemble blisterovye caps. Large spherical education ideal shape surrounded by many smaller ones. Also visible are the dark areas with less frequent placement of small globular structures.

This effect was more pronounced for $n = 10$. The dimensions of larger blocks decreased from 10-40 μ for $n = 2$ to 2-10 μ for $n = 10$. The shape of blocks also changed; they became more rounded without sharp angles (the result of melting). Using atomic-force microscopy we showed that the formation of the block structure in steel 12H15G9ND was accompanied by "layering" of the surface increasing as n increased up to 10 (Figure 2).

AFM analysis of the results of experiments on processing of 12H18N10T steel showed that unlike 12H15G9ND steel the height of columnar crystals formed in 12H18N10T steel in the direction perpendicular to the surface, even after double treatment, was much higher than that for steel 12H15G9ND. A correlation between "layering" of the steel 12H15G9ND surface and migration of columnar crystals was established: as the number of treatments increased their location shifted to the grain boundaries (blocks). A characteristic feature of the structure of the steel 12X18H10T surface is the migration of crystallites to the center, their expansion and merging causing smoothing of the surface. In this research we studied the influence of the number of pulses (treatments) on the structure of the polycrystalline steel layer and registered a decrease in the average crystallite size with the increase in the energy density and the number of pulses.

This result shows that pulsed plasma is able to control the structure of the modified layer, and, hence, its physical properties. It was found that the microhardness of the samples increased (by 150-300 MPa), which is indicative of hardening. Nonuniform changes in the surface microhardness are caused by the redistribution of the columnar crystallites and decrease in their size (height) in case of their migration to the center. The average microhardness of the surfaces of steels 12H15G9ND and 12X18H10T is much lower than the values obtained in [1, 2]. It may be caused by the formation of the block structure characteristic of plasma etching. Thus, we can conclude that for some parameters of plasma treatment, the modification of the surface structure of structural steels by melting is accompanied by the destruction of crystalline bonds and plasma etching [2, 3].

It should be noted that structural changes in the studied samples of 12H15G9ND steel can be caused by the formation of a new nitride phase FeN0,076 with the lattice parameter $a = 3.6263 \pm 0.0007$ Å (Table 1, Figure 3).

Diffraction patterns of the fragment (Figure 3) shows that the iron nitride line wider than the line of austenite. From this it follows that the nitride crystallite size smaller than that of austenite. A rough estimate of the crystallite size and microstrain WinFit program gives the following values: for iron nitride crystallite size equal to $L = 270$ Å, microdistortions $\varepsilon = 0{,}00290$, for austenite crystallite sizes are $L = 1900$ Å, microdistortions $\varepsilon = 0{,}00160$. In a comparison between the intensities of diffraction lines and austenite iron nitride for the same planes can be concluded

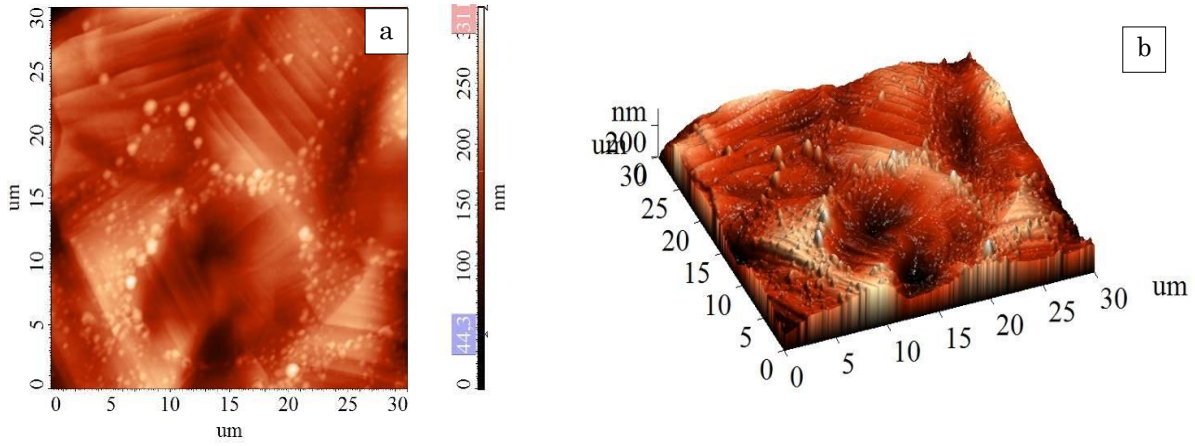

Fig. 2 – AFM image of the 12H15G9ND steel surface after tenfold treatment ($P = 13.3$ Pa, $U = 20$ kV, $Q = 15.7 \times 10^4$ J/m²)

Table 1 – Radiometric data for steel 12H15G9ND after treatment ($P = 13.3$ Pa, $U = 20$ kV, $Q = 15.7 \times 10^4$ J/m², $n = 10$)

State of the sample	n	Phase		a, Å
Untreated	0	Single phase	(Fe,C) austenite	3.6057 ± 0.0006
Treated	10	Multiphase	(Fe,C) austenite	3.5958 ± 0.0006
			(FeN0,076) iron nitride	3.6263 ± 0.0007

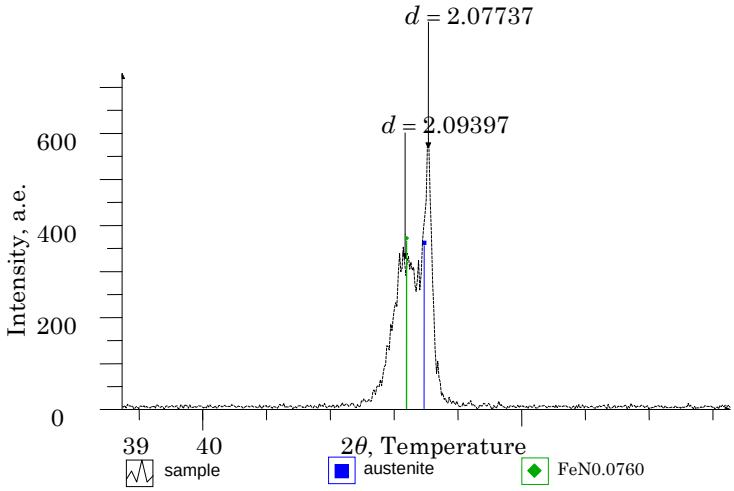

Fig. 3 – Fragment of a pattern of steel 12Х15Г9НД after processing (P = 13.3 Pa, U = 20 kV, Q = 15.7 × 10⁴ J/m², n = 10) in the interval 39°-48° 2θ

that the nitride is not a predominant phase. It is possible that iron nitride is in the surface layer, and austenite is somewhat deeper. In this case, the thickness of the nitride is small.

The nitride crystallites (L = 270 Å) are smaller than the austenite crystallites (L = 1900 Å). Comparing the data of PCA analysis for the 12H15G9ND steel with those for the 12X18H10T steel one can detect common features and differences in their structure after treatment.

The x-ray analysis shows that after 10 treatments the size of austenite crystallites decreases 4-fold as compared to the untreated steel 12X18H10T. It should be noted that after 10 treatments both phases have the same size of crystallites. Hence, repeated pulsed plasma treatment is very effective not only for grinding of 12H15G9ND steel but also for grinding of austenite crystallites, particularly, iron nitride. The x-ray analysis confirms possible increase in the microhardness of the material after surface treatment by pulsed plasma. The surface hardening can be caused by the formation of a new phase FeN0, 076 in the studied steel as it was supposed in earlier works [1, 4].

The results of our research enable us to conclude that the pulsed plasma treatment generates changes in physical and mechanical properties at the nanoscale, which may be caused by structural and phase changes and formation of defects. Furthermore, PEM analysis has shown that plasma treatment causes plasma etching, formation and redistribution of nanoscale crystallites.

REFERENCES

1. A.M. Zhukeshov, A.T. Gabdullina, *Bulletin of the NNC RK* No 2, 28 (2007).
2. A.M. Zhukeshov, F.B. Baimbetov, B.M. Ibraev, *Pulsed plasma accelerators and their technological applications* (Almaty: Kazakh University: 2009).
3. A.N. Bandura, I.E. Garkusha, O.V. Byrka, V.A. Makhlai, *Proc. 9th Int. Conf. "Interaction of Radiation with Solids"* (Minsk, Belarus: 2011).
4. A.M. Zhukeshov, A.T. Gabdullina, A.U. Amrenova, *Proc. 5th Int. Scient. Conf. "Radiation-thermal Effects and Processes in Inorganic Materials"* (Tomsk, Russia: 2006).