Zhukeshov A.M., Pak S.P.,
Gabdullina A.T.,
Amrenova A.U., Shigayeva G.G.

Structure and micro hardness of iron alloys after pulse plasma flows processing

The phase structure of surfaces of steel samples, modified by pulse-plasma processing, was analyzedusingXRD and metallographic methods. It has been shown, that after pulse plasma processing under different conditions a modified structure consisting of three new phases, including austenite, ironnitride and carbide, is formed. The dependence of phase transition and micro hardness on plasma flow parameters has been studied. A sharp decrease in the dimensions of ferrite crystallites aftertheimpact of plasma flow is observed. The main purpose of hardening of carbon steel samples is formation of nano-dimension ferrite with oriented iron carbide on grains.Pulse plasma accelerators are effective sources of plasma streams. To take such plasma streams it is used in researches in thermonuclear power and in processing of materials with the purpose to improve properties of materials. PPA appeared earlier than other accelerators and made a contribution to development and use of plasma accelerators in different spheres of science and equipment. Sense of technical use of plasma accelerators that with their help it is possible to take a stream of particles the having wide range of speed and energy.

Key words: structure, surface, pulse plasma, mixed surface, micro hardness, flow.

Жүкешов Ә.М., Пак С.П.,
Ғабдуллина А.Т.,
Әмренова Ә.У., Шиғаева Г.Г.

Темір құймалардың импульстік плазма ағындарымен өңдеуден кейінгі құрылымы және микроқаттылығы

Импульсті плазмалық үдеткіштер (ИПҮ) плазмалық ағындардың тиімді көздері болып табылады. Мұндай плазмалық ағындарды алу термоядролық энергетикадағы зерттеулерде және материалдардың бетін өңдеу кезінде қасиеттерін жақсарту мақсатында қолданылады. ИПҮ басқа үдеткіштерге қарағанда ерте пайда болды және ғылым мен техниканың әртүрлі аймақтарында плазмалық үдеткіштердің дамуы мен қолданылуына зор ықпалын тигізді. Плазмалық үдеткіштердің техникалық қолданылуының маңызы, олардың көмегімен кең диапазондағы жылдамдық пен энергиясы бар бөлшектер ағынын алуға болатындығы. Импульстік плазмамен өңдеуден өзгерген темір үлгілердің беттерінің фазалық құрылымы XRDді және металлографтық әдісті қолдану арқылы талданды. Бұл, әртүрлі шарттарда импульстік плазмамен өңдеуден кейін аустенит, темір нитриті және карбидті қосқандағы үш жаңа фазадан тұратын өзгерген құрылым түзілетінін көрсетті. Ағынның плазмалық параметрлеріндегі фазаның және микроқаттылықтың ауысуының тәуелділігі зерттелді. Плазмалық ағынмен әсерлесуден кейін феррит кристалдарының мөлшерлерінде күрт төмендегені байқалады. Көміртекті темір үлгілерді берік қылудың басты мақсаты ферриттің түйірдегі темір карбидіне бағытталған нано өлшемдерін қалыптастыру.

Түйін сөздер: құрылым, бет, импульстік плазма, аралас бет, микроқаттылық, ағын.

Жукешов А.М., Пак С.П.,
Габдуллина А.Т.,
Амренова А.У., Шигаева Г.Г.

Структура и микротвердость железных сплавов после обработки потоками импульсной плазмы

Структура фазы поверхностей стальных образцов, измененных обработкой импульсной плазмы, была проанализирована используя XRD и металлографические методы. Это показало, что после импульсной плазмы обрабатывающей при различных условиях, измененная структура, состоящая из трех новых фаз, включая аустенит, нитрит железа и карбид, изменяется. Зависимость перехода фазы и микро твердости на плазменных параметрах потока была изучена. Наблюдается острое уменьшение в размерах ферритовых кристаллитов после воздействия плазменного потока. Главная цель укрепления образцов углеродистой стали является формированием нано измерения феррита с ориентированным карбидом железа на зерне. Импульсные плазменные ускорители являются эффективными источниками плазменных потоков. Брать таких плазменных потоков используется в исследований в термоядерной энергетике и в обработке материалов с целью улучшить свойства материалов. ИПУ появился раньше чем другие ускорители и внес вклад в развитие и применение плазменных ускорителей в разных сферах науки и техники. Смысл технического применения плазменных ускорителей в том, что с их помощью можно взять поток частиц имеющий широкий диапазон скорости и энергии.

Ключевые слова: структура, поверхность, импульсная плазма, смешанная поверхность, микротвердость, поток.

UDC 50.43.15

[*]Zhukeshov A.M., Pak S.P., Gabdullina A.T., Amrenova A.U., Shigayeva G.G.

Institute of Experimental and Theoretical Physics,
al-Farabi Kazakh National University, Almaty, Kazakhstan
[*]E-mail: zhukeshov@physics.kz

STRUCTURE AND MICRO HARDNESS OF IRON ALLOYS AFTER PULSE PLASMA FLOWS PROCESSING

Introduction

The pulse plasma processing, as one of the ways of high-energy impact on surfaces of materials, is a promising method of creation of materials with given properties.This method enables us tocombinethermal influence of hot plasma withdoping by the particles of the plasma flow. Today, the most important applications of pulsed plasma processing (PPP) are: formation of p-n junctions,doping of steels with nitrogencombined with simultaneous surface recrystallization, preparation of mixed surface layers on metals and ceramics, formation of surface alloys,and a pretreatment (cleaning) of metals and ceramics for the PVD coatings [1-4].

One of the most important technological applications ofthePPP process is hardening of metal surface.In [5]the authors presented the results of investigations of properties of the modified surface layer treated by nitrogen plasma flows, with parameters varied in a wide range (several devices were used): time duration τ = 1-100 μs, plasma flow energy density E = 2-30 J/cm^2, energy of particles W = 0.4-10 keV. Based on the experimental results,theauthors concluded that changes in the surface structure do not noticeably depend onchanges in particle energy and pulse duration, but depend onthe energy density applied to the sample surface.However, these investigations do not fully describe physicalprocessesinthe materials.

As it is shown in [6], the pulse plasma accelerator “CPA-30” with a coaxial system of electrodes provides high power supply (C=75 μF, U= 30 kV) for melting the surface of metal alloys after plasma flowtreatment. However, physical properties of processedmaterialschange only under certain regimes of plasma flow, as high density of the energy flow can cause not only improvements of properties, but also destruction of the material. Therefore, the correct choice of processing regimes plays an important role intargeted treatment of material surface.In the “continuously filling”regime [7], at constant initial gas pressure in the working chamber, the plasma density varies over a wide range, and the energy has maximum E ~50 J/cm^{2}at P = 0.05-0.1Torrof the initial gas pressure.

Thus, for metal alloys such as carbon and stainless steels, the basic result of plasma treatment is hardening. Therefore,it is necessary to study physical properties of subsurface layers andthe detailed

structure of materials. In this paper we study the influence of plasma processing regimes on changes in the structure and,hence, steel hardness. The energy density ofa one-time impact was chosen as the basic parameter of processing.

Experiment

Samples of ST-3 common steel with 0.3At$_{\%}$ Cwerestudied. The samples were processedat different energy densities of air plasma on the CPU-30 accelerator. The diameter of the plasma flow (about 8 cm)wasgreater than the dimension of samples (15×15×4 mm^3). The duration of 100-250 kA discharge current was about 4-7 μs. The X-raydiffractionanalysis (XRD) of the processed samples was carried out on "D8 Advance" diffract meter, the micro hardness was measured on the metallurgical microscope "Metaval". The lattice parameters of materials were determined by a specialized program of the diffract meter [8].

We will further consider the results of structural studies of the carbon steel treated by plasma flows in a continuous mode. In this modewe got the values of changes in the structure of materials after single and multiple plasma treatment for different values of the initial pressure of 0.04 – 0.5 Torr. Parameters of carbon steel processingaregivenin table 1.

Table 1 – The data for all carbon steel samples

Sample number	N	Energy densityQ,J/cm^2	Lattice parameter a,Å	2θ$_{max}$, degree.	I$_{max}$, abs.u.	phase	L, Å
Initial	0	-	2.8691±0.0005	44.723	699	Fe	1160
One-time treatment							
#3	1	16	3.6172±0.0047	43.280	24.2	Fe-γ	175
			2.8631±0.0004	44.798	321	Fe	730
#4	1	22	3.6141±0.0017	43.364	19.8	Fe-γ	160
			2.8622±0.0007	44.798	221	Fe	610
#5	1	32	3.6212±0.0037	43.356	26.0	Fe-γ	240
			2.8630±0.0005	44.787	176	Fe	460
#7	1	44	3.6184±0.0044	43.262	13.6	Fe-γ	110
			2.8630±0.0003	44.773	121	Fe	115
#9	1	48	3.6223±0.0028	43.244	12.7	Fe-γ	140
			2.8610±0.001	44.737	134	Fe	145
Multiple treatment							
#1	5 5	25	3.6217±0.011	43.213	32.6	Fe-γ	195
			2.056±0.001	44.005	19.9	FeC	
			2.8630±0.0004	44.751	125	Fe	380
#2	10	28	3.6254±0.0011	43.095	33.5	Fe-γ	210
			2.055±0.001	44.005	19.0	FeC	
			2.8605±0.0003	44.749	74.0	Fe	395
#3	20	26	9.0760±0.0046	39.769	7.41	$Fe_{24}N_{10}$	
			3.6217±0.0011	43.083	44.3	Fe-γ	180
			2.8607±0.0005	44.681	62.2	Fe	275
#4	30	25	9.1082±0.0046	39.877	9.78	$Fe_{24}N_{10}$	
			3.6295±0.0018	41.836	7.96	Fe-γ	145
			2.8603±0.0009	44.773	66.0	Fe	200

The basis of the initial sample of carbon steel is ferrite with a crystal lattice parameter a = 2.8691angstrom.The basic phase of the sample has coherent diffraction areas (crystallite size) of a dimension L = 1160Å.

An analysis ofthe XRD data (Figure1) shows that the single treatment causesstructural and phase transformationsofthe material. The original structure of carbon steel $Fe_{\alpha}$with the space-centered lattice and the lattice parameter a = 2.8691 ± 0.0005 Å is converted into a two-phase solution. One of the phases is a solid α-solution – ferrite, the lattice parameter is not actually changing.Pulsed plasma treatment leads to the formation of the second phase of the face centered lattice, which corresponds to the austenite (γ-Fe). Thespecificfeature of processing for all samples is the presence of anew austenite phase. As it is shown in table 1, the maximal crystallite size L of austenite is contained in the sample #5 processed by 32 J/cm^2. For example, a detailed phase diagram of the processed sample #3 is given in Figure 1.The obtained results show that the influence of the plasma flow on the structural parameters does not have a trivial character. The dimension of ferrite crystallites strongly depends on plasma energy varies in the range 40-50 J/cm^2. The austenitic phase reaches a weak maximum at an energy densityof 32 J/cm^2, when the maximal crystallite quantity and maximal dimensions of austenite crystallites are observed.

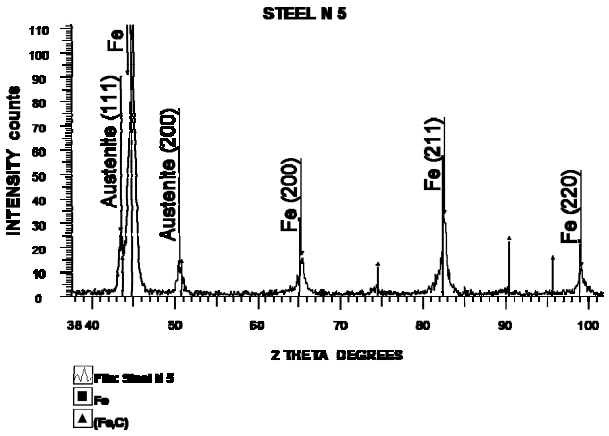


Figure 1 – The X-ray diffraction patternof sample #5 after a single treatment

We will consider the multipleplasmaimpact on the surface of this material.When the surface of carbon steel is treated by several plasma pulses at P = 0.1 Torr, a further increase in the intensity of formation of γ-Fe is observed (Figure2).

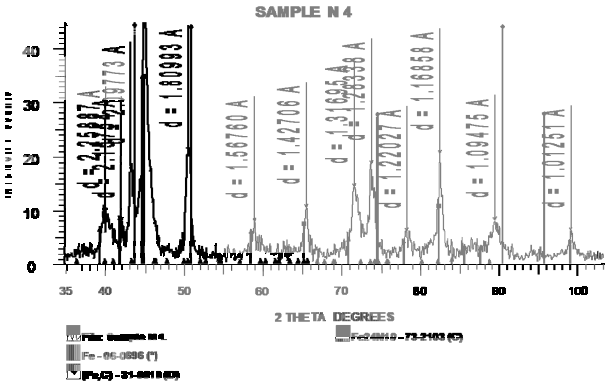


Figure 2 – The X-ray diffraction pattern of sample #4 treated 30times

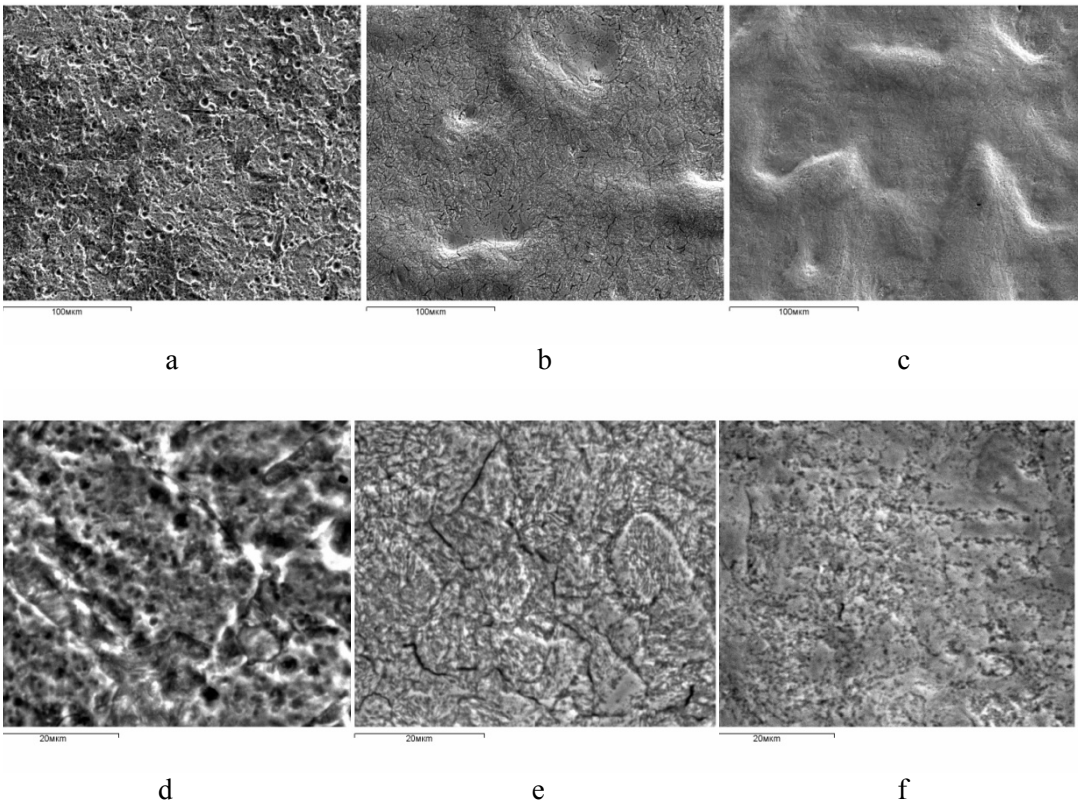
Processing of the 5-th and 10-th pulses in the two-phase solution of α-Fe (a = 2.8603 ± 0.0009 Å) and γ-Fe (a = 3.6295 ± 0.0018 Å) revealed the presence of a small amount of marten site FeC (one line 2θ = 44.0050, samples # 1, 2). Further processing of the 20-th and 30-th pulse led to formation of iron nitride $Fe_{24}N_{10}$.Nitride was observed in the diffraction broadened diffuse lines (samples#3, 4). The nitride intensity peaks in the sample number 4 treated 30 times were higher than the peaks for the sample number 3, which was treated 2 times by 20 plasma pulses. In addition, the lattice parameter of iron nitride in the samples # 3,4 differed from the ideal sample: a = 9,2150 Å, which could be caused by the deformation of nitridelattice.

Table 1shows the results ofdetermination ofcrystallite sizeof ferrite andaustenite in thesteelsamplesafter processing using Scherer's method.It can be seenthatthe crystallite sizeof ferritedecreases andthe crystallite sizeof γ-Feremainsunchangedwith increasingQ,and increasingN,butin the case ofa single treatment this effect ismore pronounced.These results arein good agreementwith those obtainedbythe SEM.The changein the grain sizecan be clearly seenin the photographsof the microstructureof the surface afteretchingfor grainvisualization (Figure3), and may be caused bythe dissolutionof large grainsand increase in the number of dispersion structures (Figure 3d, e,f) at higher Q values.

The stage of formationof the twophasesolutionafter plasmatreatmentat the residualair pressurein the CPU-30chamberis clearly seenin Figure4.The figure shows that the

two-phase solution is for medasprecipitatesat the grain boundaries even at low Q (Q for the sample#1is 5J/cm^2),and the increase inthe energy densityof the plasma flowpasses through a seriesof successive stages. Phase transformation reachesits maximumaftermultiple plasma treatment.

Austenitizingpromotesheating of the material surface,which is also typical ofelectronic beam processing. As a result,a large amount ofthe carbidesource materialis dissolved andtransformedinto austenitewith high carbon content. The surface profileof the carbon steel sample after plasmatreatmentas obtainedby atomic forcemicroscopy (Figure5). In Figure 6 acolumnargrainstructure is observed, which is characteristic of the formation of FeC phase.

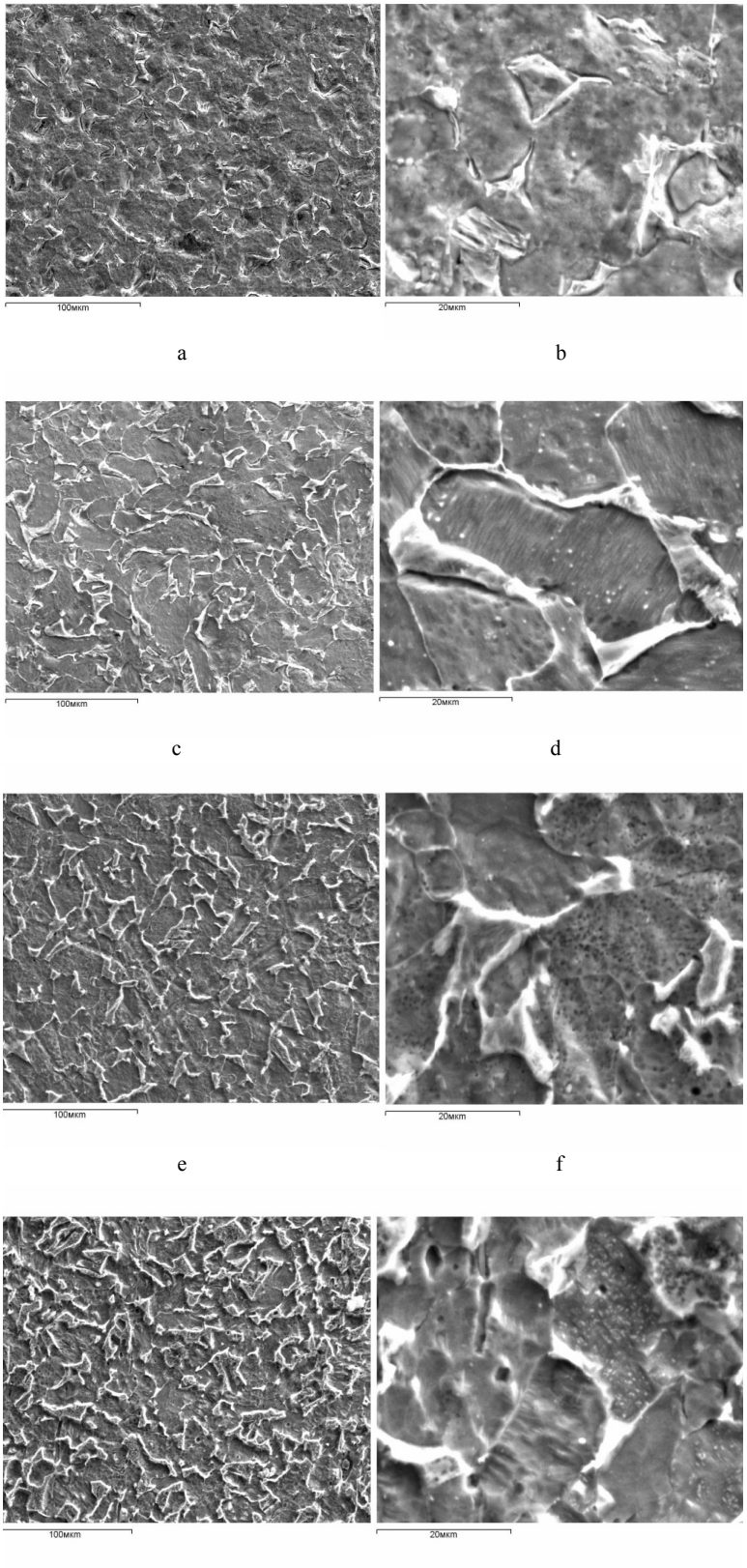


a,b, c- × 400, d, e, g – ×2000 sample number 3, 4, 5, respectively

Figure 3 – The structure of the surface of steel samplesafter air plasma treatmentatP = 0.04Torr, N = 1

Figure 6a shows the influence of the sample structure on the micro hardness of the material. The micro hardness of the processed samples changes proportionally to the energy density, however, the sameregularity is observed. As it is shown in Figure 3a, for the first group of samples(#1,2) the value of micro hardness does not change considerably at 2000 MpA. For the second group (samples # 3,4,5,6) the micro hardness increases two-fold (4000 MPa), and for the third group (samples #7,8) it increases by more than three-fold (7000 MPa).However, only the third group is of practical interest because it has high hardness in spite of greater crystallite dimensions. In general, the proportionality of the energy is observed for all samples.Thus, there is an optimal value of energy density in area 20-44 J/cm^2, at which the micro hardness of carbon steel is rather high ~ 4000 MPaand crystallites with minimal size 115 nm are formed.At low energy below20 J/cm^2 no hardening is observed, and at highenergies crystallite sizes increase.

HV measurements of the carbon steel surface after repeatedtreatmentshowed that thefirst 5of 10pulsesincreased hardness, and further processing stabilized the hardening process (Figure 6b) due to completion of the austenitization and formation of the marten site phase andiron nitride.



a, c, e, g – surface structure×400, b, d, f, h ×2000

Figure 4 – Formation ofa two-phase solution after plasma treatment (P= 0.5 mm Hg, N = 1, samples number 1, 2, 3, and 4, respectively)

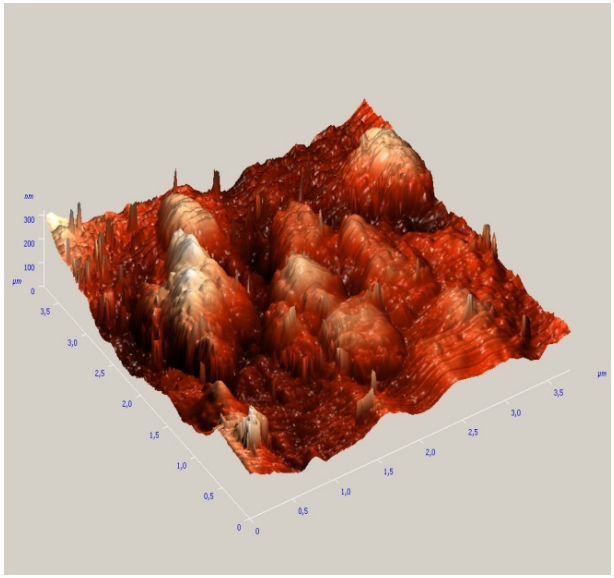
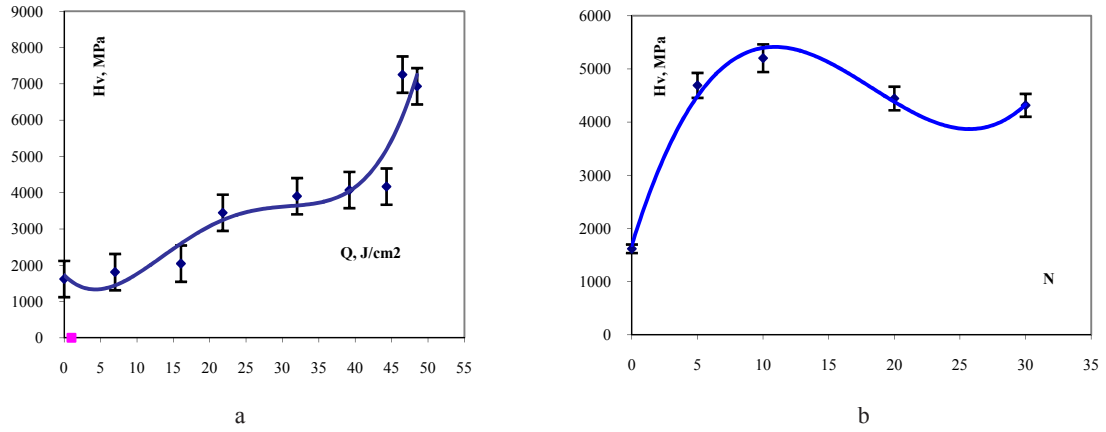


Figure 5 – A surface profile of carbon steel sample after plasma treatment



a b

Figure 6 – Micro hardness as a function of energy density for single (a) and multiple (b) processed samples

Conclusion

The influence of plasma processing regimes on changes in the structure and, as a consequence, on steel hardness is studied. Under the action of plasma flows,physical and mechanical properties of common steel change differently. In case ofa single treatment,hardeningof the material is proportional to the energy density, but after 5 pulses the saturation stage is reached. The single treatmentcausesstructural and phase transformations of the material. The original structure of common steel $Fe_{\alpha}$with the volume centered lattice is converted into a multiphase solution. A significant feature of processing for all samples is the presence of a new austenitephase at energiesup to 40 J/cm^2 and a nitride phaseat higher energies and multiple treatments. At high speed crystallization process a large amount ofcarbideis dissolved andtransformedinto austenitewith high carbon content. In the AFM pictures a columnar-grainstructure is observed, which is indicativeofthe formation ofthe high carbide (martensite) phase, formed due to quick cooling of the material.The carbon atoms, probably, diffusion from material bulk to surface and participate in new phase creation process.

The crystallite sizeof ferritedecreases, andthe crystallite sizeof γ-Fedoes not change with the increase in energy and number of processing N, butin the case ofa single treatment it ismore pronounced.

The changeofthe grain size may be caused bythe dissolutionof large grainsand higherdispersion. It is shown that the two-phasesolution is formedin the form ofprecipitatesat the grain boundaries. Phase transformation reaches its maximum after multiple plasma treatments. The micro hardness of the material also depends on its structure. The micro hardness of processed samples changes proportionally to energy density, however, at multiple treatment it reached its maximum after 5 treatments.

The refore we an conclude that grinding grains of crystals are associated with the surface hardening of steels. Small grains are formed as a result of fast cooling (at speed of 10^8 – 10^9 K/s) of the subsurface area and heat dissipation throughout the material.

References

1 Chebotarev V.V., Garkusha I.E., Bovda A.M.. atal. Application of pulsed plasma accelerators for surface modification // Nukleonika. – 2001. – Vol. 46. – P.27-30.

2 Peng Z., Miao H., Wang W. Hard and wear-resistant titanium nitride films for ceramic cutting tools by pulsed high energy density plasma // Surf. Coat. Tech. – 2003. – Vol.166. – P.183-188.

3 Tereshin V.I., Bandura A.N., Byrka O.V. at al. Surface Modification and Coatings Deposition under Plasma Streams Processing// Adv. Appl. Plas. Sci. – 2003. – Vol.4. – P.265-270.

4 Anishik V.M., Uglov V.V. Modification of instrumental materials by ion and plasma beams. – Minsk.: BGU, 2003. – 191 p.

5 Chebotarev V.V., Garkusha I.E., Langneret J. at al.Surface Structure Changes Induced by Pulsed Plasma Streams Processing// Prob. of Atom. Sci. and Tech. Series: Plasma Physics. – 1999. – № 3(3). – P. 273-275.

6 Baimbetov F.B., Zhukeshov A.M., Amrenova A.U. Dynamics of plasma flow formation in a pulsed accelerator operating at a constant pressure // Tech. Phys. Lett. – 2007. – Vol.33. – P. 77–79.

7 Zhukeshov A.M. Plasma flow formation in a pulse plasma accelerator in continuos filling regime// Plasma Dev. Oper. – 2009. – Vol.17. – P.73–81.

8 Zhukeshov A.M., Gabdullina A.T., Pak S.P. Modification of materials with particle beams and plasma flows // Proc. of 10–th Inter. Conf. Tomsk. – 2010. – P.376-377.

References

1 Chebotarev V.V., Garkusha I.E., Bovda A.M.at al. Nukleonika. 46.(2001). – P. 27-30.

2 Peng Z., Miao H., Wang W.. Surf. Coat. Tech.166. (2003). – P. 183-188.

3 Tereshin V.I., Bandura A.N., Byrka O.V. at al. Adv. Appl. Plas. Sci.**4.**(2003). – P. 265-270.

4 Anishik V.M., Uglov V.V. Modification of instrumental materials by ion and plasma beams. – Minsk: BGU, (2003), 191 p.;

5 Chebotarev V.V., Garkusha I.E., Langner J. at al.Prob. of Atom. Sci. and Tech. Series: Plasma Physics. № 3(3). (1999). – P. 273-275.

6 Baimbetov F.B., Zhukeshov A.M., Amrenova A.U. Tech. Phys. Lett**.**33. (2007). – P. 77–79.

7 Zhukeshov A.M. Plasma Dev. Oper.17. (2009). – P. 73–81.

8 Zhukeshov A.M., Gabdullina A.T., Pak S.P. Modification of materials with particle beams and plasma flows // Proc. of 10–th Inter. Conf. Tomsk. (2010). P.376-377.