doi: 10.56761/EFRE2022.C2-P-956001

Study of the influence of plastic deformation of the HSS M2 surface on ion-plasma nitriding in the glow discharge

R. Vafin, A. Asylbae, D. Mamontov, I. Sklizkov*

Ufa State Aviation Technical University, Ufa, Russia
alexander.aslb@gmail.com

Abstract. This paper examines the influence of plastic deformation of the HSS M2 surface on the characteristics of the hardened layer after ion-plasma nitriding in the glow discharge. In the study it was found that the plastic deformation of the steel surface increases the rate of nitrogen diffusion deep into the material, due to an increase in dislocation density and the formation of microdefects, due to a highly refined grain-like ultrafine grain structure. It was also found that the plastic deformation of the steel surface before ion-plasma nitriding leads to a 2-fold increase in the thickness of the hardened layer HSS M2, due to an increase in surface free energy, which contributes to increased adsorption of the saturation element and the formation of nitrides in the near-surface layer of nitrided material.
Keywords: plastic deformation, ion-plasma nitriding, glow discharge, ultrafine grain.

1. Introduction

Due to the constant development of science, technology and engineering, ever higher requirements are imposed on the materials used from the point of view of mechanical and performance properties. This necessitates the use of expensive materials with the required set of properties or the structural modification of already used materials by thermal or mechanical treatment [1–5].

One of the effective methods of heat treatment in mechanical engineering is the technology of ion-plasma nitriding. This is mainly due to the advantages that differentiate it from similar methods. Due to ion-plasma nitriding, machined parts and tools have enhanced mechanical and operational properties: strength, hardness, wear resistance, scratch resistance, corrosion resistance, fatigue resistance and heat resistance [6].

It is known that diffusion of atoms in metals is greatly influenced by various structural defects - deviations of the lattice structure from the ideal one [7]. As the structural defects increase, the diffusion rate in the metal increases. However, along with structural defects, diffusion is also affected by the size of the metal grain: the finer the grain, the higher the diffusion rate [8, 9]. Therefore, to increase the diffusion rate in metals, methods of plastic deformation have recently become increasingly widespread. One of these methods is the method of intense plastic deformation by torsion, which consists in deformation of metal by two simultaneously acting forces: compression and torsion (Fig.1a).

The purpose of this work was to study the influence of plastic deformation of the HSS M2 surface on the characteristics of the hardened layer after ion-plasma nitriding.

2. Research methodology

HSS M2 samples were taken for the experiment (Table 1).

Table 1. Chemical composition of HSS M2

C, %	Si, %	Mn, %	Ni, %	Cr, %	Mo, %	W, %	V, %	Co, %
0.82–0.9	max 0.5	max 0.5	max 0.4	3.8–4.4	4.8–5.3	5.5–6.5	1.7–2.1	max 0.5

To study the effect of plastic deformation of the HSS M2 surface on the characteristics of the hardened layer after ion-plasma nitriding, half of the studied samples were subjected to severe plastic deformation by torsion. This method of treatment consists in mechanical deformation of the metal by

two simultaneously acting forces – compression and torsion, which results in a highly refined grain-like structure – UFG (ultrafine grained) (Fig.2b) [8, 9].

In this work, the samples before ion-plasma nitriding were subjected to 43% cold settling and 1.5 turns of torsion at a hydrostatic pressure of 4 GPa on the " SKRUJ-200" unit, according to the scheme in Fig.1a.

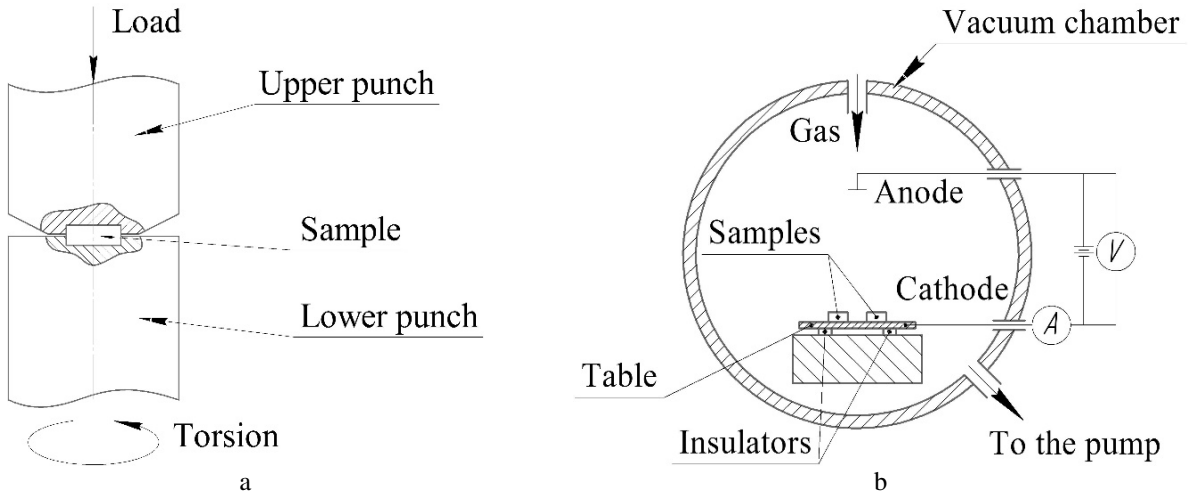


Fig.1. Process schemes: a – severe plastic deformation by torsion, b – ion-plasma nitriding in a glow discharge.

Ion-plasma nitriding in the glow discharge was carried out on the modernized ELU-5M unit (Fig.1b). Before treatment in a vacuum chamber, ionic cleaning of the sample surface was carried out for 15 min in an argon atmosphere at a pressure of p = 10–20 Pa. The ion-plasma nitriding was carried out in a gas mixture of argon, nitrogen and hydrogen (50 % Ar+35 % N_2+15 % H_2) at gas pressure p = 200 Pa and temperature T = 450 °C for 2, 4 and 6 hours (Table 2).

Table 2. Conditions of ion-plasma nitriding

№	Temperature, °C	Pressure, Pa	Time, h
1			2
2	450	200	4
3			6

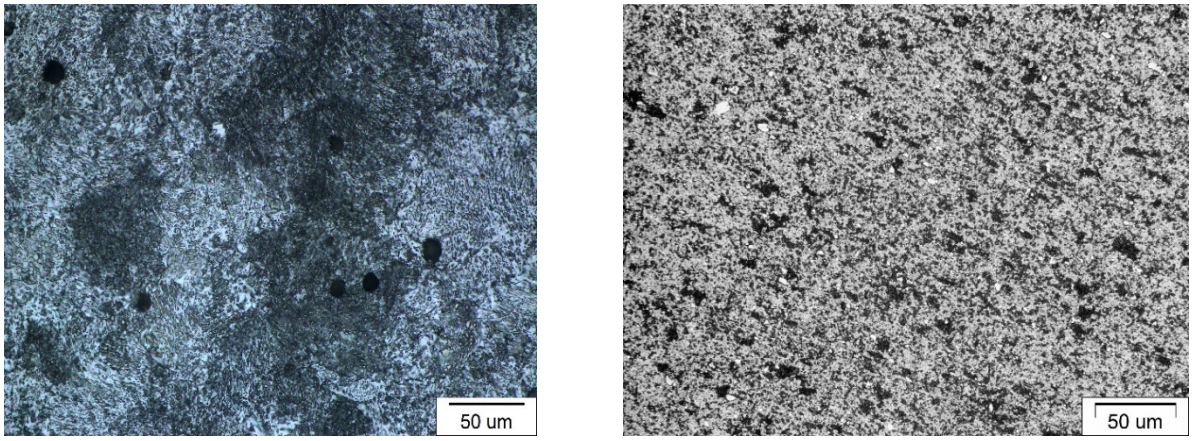
The microhardness of the surface layer of the samples after ion-plasma nitriding was investigated by the Vickers method on oblique sections (angle ~7°) using an automatic hardness meter EMCO-Test DuraScan 50. To reveal the microstructure, the studied samples were chemically etched for 10 s with a solution of acids: C_2H_5OH (80 ml), HNO_3 (10 ml), HCl (10 ml) and $C_6H_3N_3O_7$ (1 g). The thickness of the nitride zone was estimated from the obtained optical images of the microstructures using microscope Olympus GX51.

3. Results and discussion

To study the influence of plastic deformation (PD) of the HSS M2 surface on the characteristics of the hardened layer after ion-plasma nitriding (IPN) in the glow discharge (GD), two groups of samples were taken: the first group was annealed at 850 °C (Fig.2a), the second group was subjected to severe plastic deformation by torsion (SPDT) (Fig.2b).

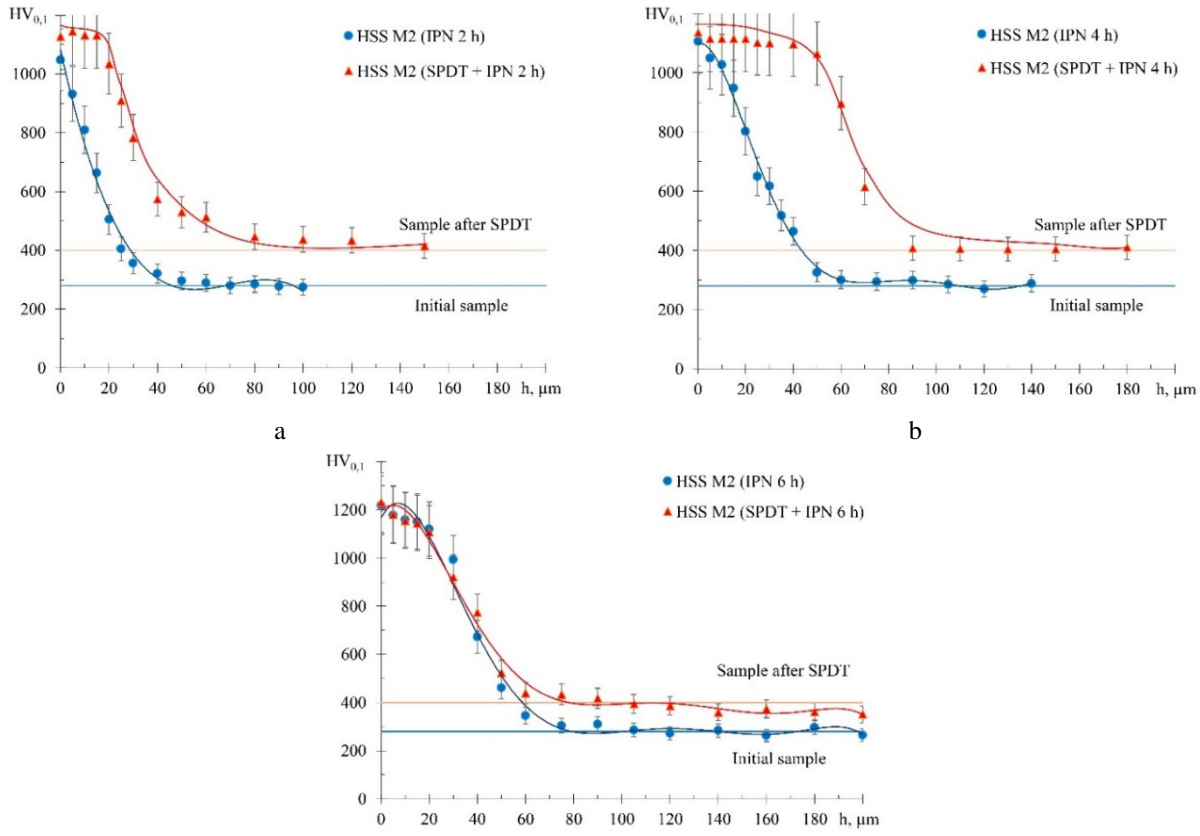
From the images of the microstructure (Fig.2a and Fig.2b) we can see how the structure of the annealed sample (Fig.2a) of the HSS M2 after SPDT (Fig.2b) has changed. As a result of PD of the HSS M2 by two simultaneously acting forces – compression and torsion, the steel structure was

deformed, forming a highly refined grain-like structure – UFG (ultrafine grained) [8, 9]. This structure is characterized by increased physical-mechanical and performance properties, in particular: diffusion rate, surface and volume hardness, wear resistance.



a b

Fig.2. Optical images of the HSS M2 microstructure: a – initial sample, b – SPDT sample.



a b

c

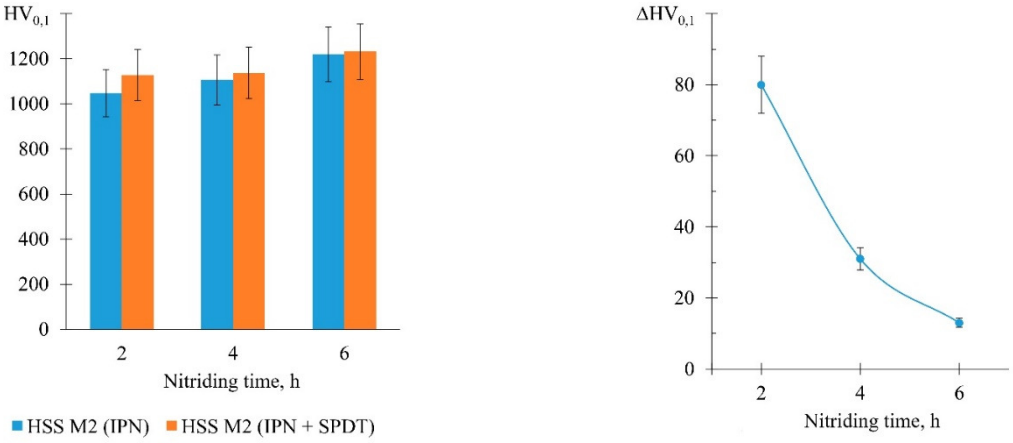
Fig.3. Graphs of microhardness depth distribution at different nitriding times: a – 2 hours, b – 4 hours, c – 6 hours.

Fig.3 shows graphs of microhardness depth distribution of samples after IPN in the GD at different nitriding times.

The graphs show that with increasing duration of IPN the surface hardness and thickness of the hardened layer of the initial sample increases in ~ 1.2 and 1.5 times respectively, at SPDT sample the

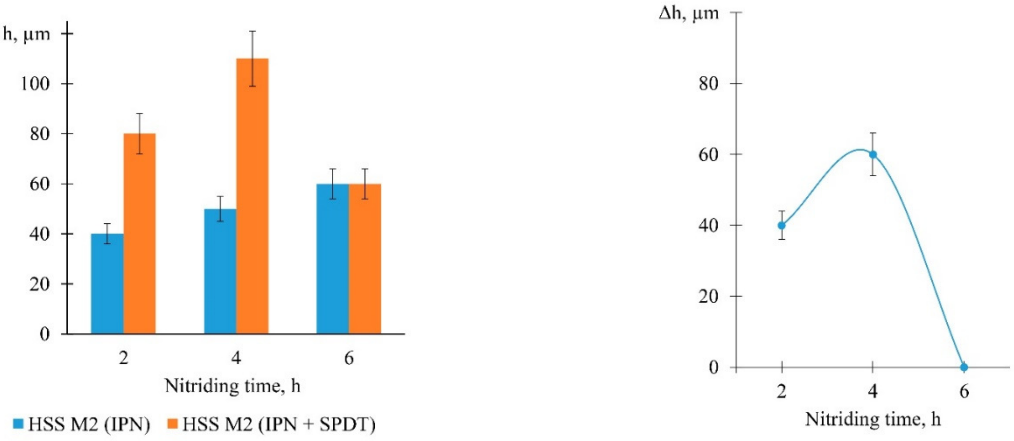
surface hardness practically does not change and is in the near-surface zone (0–20 μm) ~ 1150 HV, and the thickness of the hardened layer increases in 2 times. From the presented graphs it is also clear that the application of SPDT before IPN contributes to an increase in the surface hardness and thickness of the hardened layer of the samples by 1.5 times compared to the initial samples.

For a more detailed analysis of the results obtained, we prepared graphs of dependences of surface hardness (Fig.4) and thickness of the hardened layer on nitriding time (Fig.5).



a b

Fig.4. Graph of dependence of surface hardness of the hardened layer on the nitriding time: a – absolute values of surface hardness, b – hardness increment in the SPDT sample relative to the initial sample.

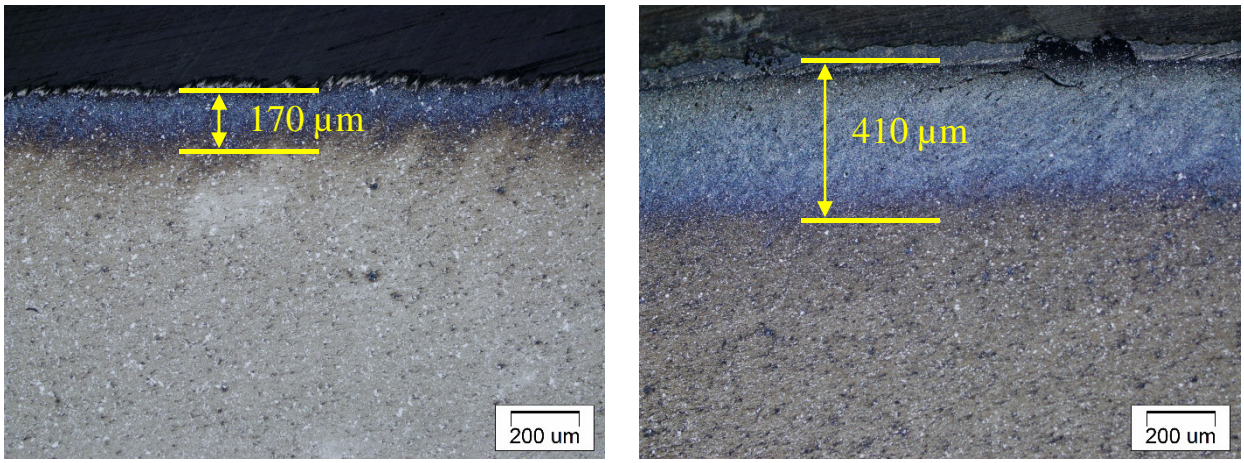


a b

Fig.5. Graph of dependence of thickness of the hardened layer on nitriding time: a – absolute values of thickness, b – thickness increment in the SPDT sample relative to the initial sample.

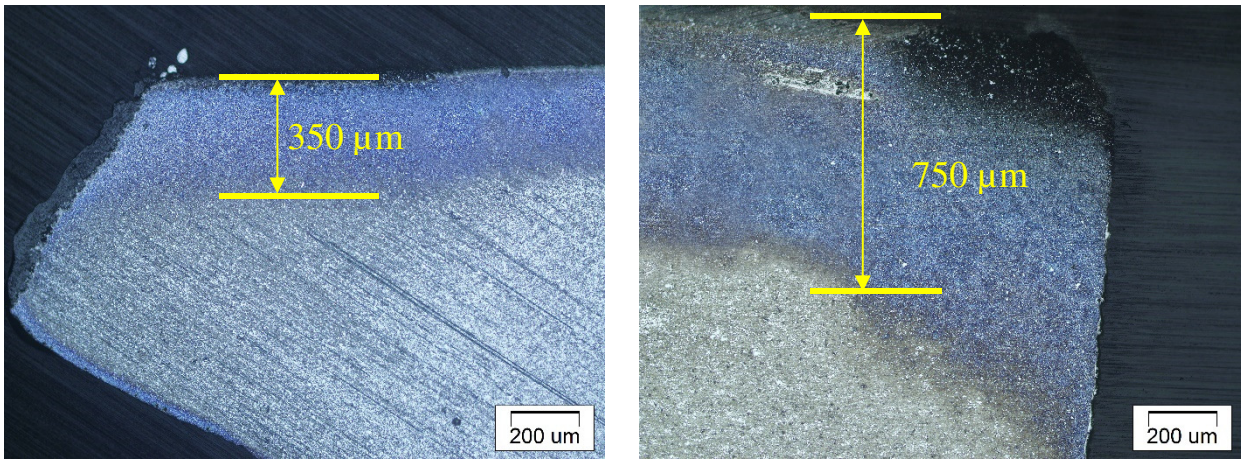
Fig.4 shows that the influence of PD of the steel surface on the value of surface hardness is minimal – the maximum increase is 7.6% at IPN for 2 hours. However, the PD of the steel surface has a significant effect on the thickness of the hardened layer. At IPN for 4 hours the growth of the hardened layer in the SPDT sample, relative to the initial sample, amounted to 110%. However, at the IPN for 6 hours there is no influence of PD on the characteristics of the hardened layer. This is due to the fact that in the first hours of nitriding the surface layer of the material is maximally saturated with nitrogen, and the nitrides formed during the following nitriding prevent further nitrogen diffusion deep into the material.

Fig.6–8 show images of the microstructures of the samples after IPN in the GD at different nitriding times.



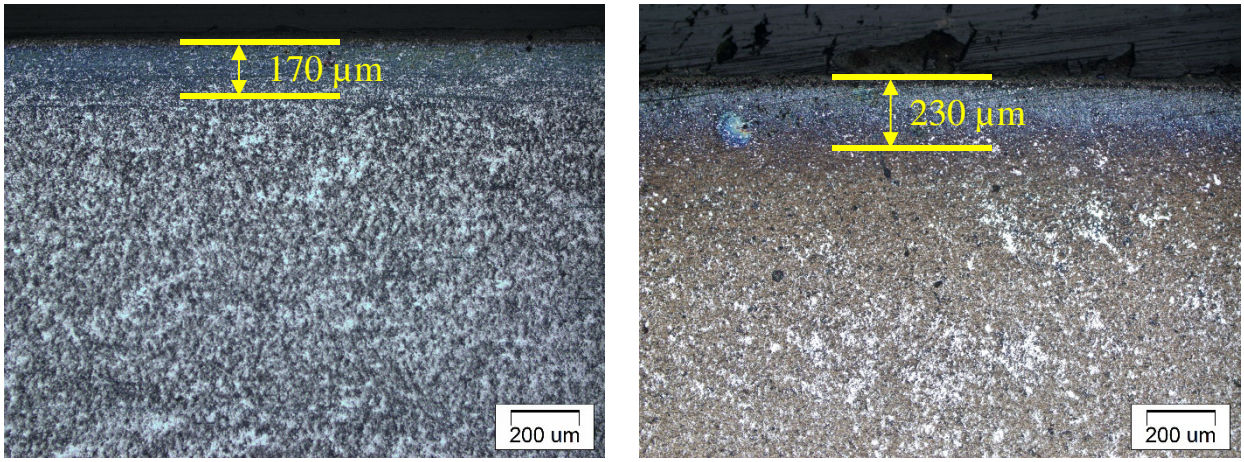
a b

Fig.6. Optical images of the HSS M2 samples microstructure after IPN at 450 °C for 2 hours: a – initial sample, b – SPDT sample.



a b

Fig.7. Optical images of the HSS M2 samples microstructure after IPN at 450 °C for 4 hours: a – initial sample, b – SPDT sample.



a b

Fig.8. Optical images of the HSS M2 samples microstructure after IPN at 450 °C for 6 hours: a – initial sample, b – SPDT sample.

Since the study of samples was carried out on oblique sections with an angle of ~7°, the values of the thicknesses of the nitride zone from the images of the microstructures should be divided by 10. Therefore, the true values of the thicknesses of the nitride zone for each of the samples will correspond to the values from Table 3.

Table 3. True values of the thicknesses of the nitride zone of samples after IPN in the GD at different nitriding times

Time, h	Thickness of the nitride zone, μm	
	Initial sample	SPDT sample
2	17	41
4	35	75
6	17	23

From the images of microstructures presented above (Fig.6–8) it is clear that the thickness of the nitride zone in SPDT samples, after IPN, is two times greater relative to the initial samples, which corresponds to the results of microhardness measurements. However, in the images of samples nitrided at 6 hours, the difference in the thickness of the nitride zone is almost absent, which also corresponds to the results obtained above.

Such results are explained by the fact that the SPDT samples, after PD by torsion, have formed a highly refined grain-like UFG structure [9]. Such a structure leads to an increase in surface free energy [10], which contributes to an increase in adsorption of the saturating element and formation of nitrides in the near-surface layer of the nitrided material, and leading to an increase in hardness in the near-surface zone (0–20 μm). Also, due to increased dislocation density and the formation of microdefects and grain refinement, the diffusion rate of the saturating element deep into the material increases [7–9], which contributes to an increase in the thickness of the hardened layer (Fig.4 and Fig.5).

4. Conclusion

As a result of the study, it was established that:

- Plastic deformation of the steel surface increases the rate of nitrogen diffusion in the HSS M2, due to the increased dislocation density and the formation of microdefects, due to a highly refined grain-like ultra-fine grain structure.
- Plastic deformation of the steel surface before ion-plasma nitriding leads to an increase in the thickness of the hardened layer of the HSS M2 ~ 2 times, due to an increase in surface free energy, increasing the adsorption of the saturation element and the formation of nitrides in the near-surface layer of nitrided material.
- At ion-plasma nitriding for 6 hours there is no influence of plastic deformation on the characteristics of the hardened layer, due to oversaturation of the surface layers of the material in the first hours of nitriding, and because of the nitrides formed in the process, which prevent further diffusion of nitrogen deep into the material.

Acknowledgement

This work was supported by a grant in the form of subsidies in science from the budget of the Republic of Bashkortostan for state support of young scientists – postgraduate students and candidates of science (theme: AT-TM-04-21-GB).

5. References

[1] Zhengjie Lin, Liqiang Wang, Kelvin Wai Kwok Yeung, Jining Qin, *SOJ Mater. Sci. Eng.*, **1**(1), 01, 2013; doi: 10.15226/sojmse.2013.00101

[2] Kyun Taek Cho, Kyung Song, Sang Ho Oh, Young-Kook Lee, Won Beom Lee, *Surf. Coat. Technol.*, **232**, 912, 2013; doi: 10.1016/j.surfcoat.2013.06.123
[3] Lie Shen, Liang Wang, Yizuo Wang, Chunhua Wang, *Surf. Coat. Technol.*, **204**(20), 3222, 2010; doi: 10.1016/j.surfcoat.2010.03.018
[4] Ke Lu, Jian Lu, *J. Mat. Sci. Technol.*, **15**(3), 193, 1999; url: https://www.jmst.org/EN/Y1999/V15/I03/193
[5] Ke Lu, Jian Lu, *Mat. Sci. Eng., A*, **375**, 38, 2004; doi: 10.1016/j.msea.2003.10.261
[6] Arzamasov B.N., Bratuhin A.G., Eliseev YU.V., Panajoti T.A., *Ion chemical heat treatment of alloys*. (Moscow: The Bauman Moscow State Technical University, 1999).
[7] Bokshtejn B.S., *Diffusion in metals*. (Moscow: Metallurgy, 1978).
[8] Valiev R.Z., Zhilyaev A.P., Langdon T.G., *Bulk nanostructured materials. Fundamental and Applications*. (Hoboken: Wiley/TMS, 2014).
[9] Valiev R.Z., Aleksandrov I.V., *Nanostrukturnye materialy, poluchennye intensivnoj plasticheskoj deformaciej*. (España: Universidad Complutense de Madrid, 2000).
[10] Li D., Wu J., Miao B., Zhao X., Mao C., Wei W., Hu J., *Surf. Eng.*, **36**(5), 524, 2020; doi: 10.1080/02670844.2019.1641953