

Changing the structure and phase states and the microhardness of the R6M5 steel surface layer after electrolytic-plasma nitriding

B.K. Rakhadilov*, Sh.R. Kurbanbekov, M.K. Kilishkhanov,
A.B. Kenesbekov

S. Amanzholov East Kazakhstan State University, Ust-Kamenogorsk, Kazakhstan

E-mail: rakhadilovb@mail.ru

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The article examines the changes of the structural-phase states and the microhardness of the R6M5 steel surface layer after electrolytic-plasma nitriding. It is found that after electrolytic-plasma nitriding of the R6M5 steel surface, diffusion layer is formed, which is a nitrogen martensite. The phase composition of the diffusion layer varies depending on the nitriding temperature. An increase of R6M5 steel microhardness, depending on structural-phase state, is found out. The main factor, influencing the increase of microhardness of R6M5 high-speed steel with electrolytic-plasma nitriding, is the formation of nitrogen martensite with monophasic nitride Fe_4N (γ' - phase), as well as the formation of fine inclusions, hardening phases in the surface layers.

Keywords: microstructure, microhardness, nitriding, phase composition, high-speed steel.

Introduction

Performance of cutting tools is largely determined by the surface layer [1]. To increase the hardness, strength and wear resistance of the cutting tools, various methods for chemical-thermal treatment (namely, the nitriding) are widely used. In recent years, the increasing development and use belongs to the methods of plasma nitriding that allow you to avoid the drawbacks of traditional methods

of nitriding [2]. Plasma nitriding provides formation of nitrided layer with a given structure [3-5] on the surface of the workpieces. One of the promising methods for plasma nitriding, which allows to significantly reduce the overall processing time, as well as significantly increase the hardness and wear resistance of steels, is electrolytic-plasma nitriding [6,7]. When electrolytic-plasma nitriding, significant changes in structural and phase states are undergoing, and accordingly, changes of the material properties in thin surface layers due to physical effects of low-temperature plasma ions and electric discharge. Developing restructuring processes, structural and phase transformations occur in conditions, far from thermodynamic equilibrium states, and they let you receive the modified surface layers with a unique set of physical and mechanical properties [8].

In connection with the above, the purpose of this paper is to study changes in the structural and phase states and the microhardness of the R6M5 steel surface layer at the electrolytic-plasma nitriding.

Material and research methods

In accordance with the purpose, R6M5 high-speed tungsten-molybdenum steel was chosen as an object of study. The choice of material is based on the fact that the R6M5 steel is the most widespread in the metal processing, and as it is the typical high-speed steel of moderate heat resistance.

Workpiece samples for research have been cut from bars of R6M5 steel in the form of parallelepipeds with dimensions (10x30x30) mm³. The samples were then subjected to the standard steel heat treatment: quenching up to 1230 °C in oil, and subsequent tempering (three times) at 560 °C (the period of each tempering is 1 hour; cooling is in air) [9]. After heat treatment, samples have been grounded and polished, and then were subjected to electrolytic-plasma nitriding.

Electrolytic-plasma nitriding of the samples was performed on a pilot plant [10], having the following main parts: an electrolytic cell, power supply, automatic control system, cooling system of the electrolyte, the electrolyte supply system (Figure 1). The process for electrolytic-plasma nitriding was the process of processing the R6M5 steel sample in glow-spark discharge (low-temperature plasma), set up between the electrolyte and the sample surface. To create and to maintain low temperature plasma, there was used an aqueous solution electrolyte, containing 20 % of carbamide and 10 % of sodium carbonate, which is a ion source for the nitriding process. Processed sample have been half immersed to the open reactor (electrolytic cell), filled with electrolyte. At that, the electrolyte was in circulation mode. Rectified voltage was supplied to the sample (cathode) and the solid electrode (anode). When voltage was applied near the surface of the sample, the gas-vapor shirt consisting of electrolyte vapors was created; this "shirt" contained ions of the electrolyte, and, respectively, the nitrogen ions. It was a low-temperature plasma, actively saturating atmosphere for the nitriding.

The processing was carried out in an aqueous solution electrolyte, containing 20% of carbamide and 10% of sodium carbonate in the following mode: samples nitriding temperature - $T = (450-550) ^\circ\text{C}$, the applied voltage between the anode

and the sample, when heated to the nitriding temperature - 320 °C, and when exposed at the $T = (450-550) ^\circ\text{C}$ - 200 V, nitriding time - 7 minutes. Nitriding temperature was chosen below the tempering temperature, which ensures the hardness of the matrix at baseline.

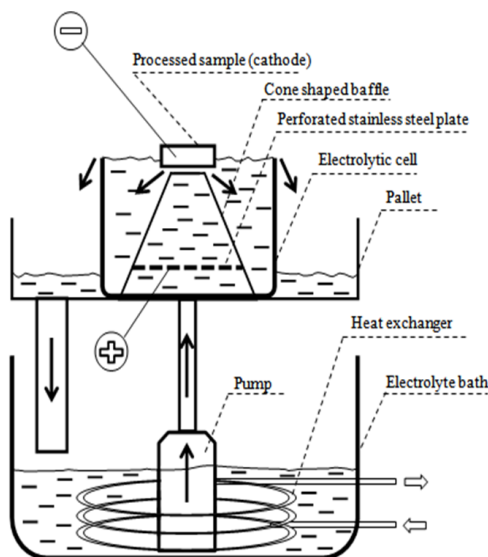


Figure 1. The scheme of electrolyte-plasma nitriding equipment.

Metallographic examination was carried out on the optical microscope "ALT-AMI-MET-1M". The microstructure of steel samples was examined in a scanning electron microscope (SEM) JSM-6390LV. Study of the phase composition of carbide particles was carried out by the EBSD-analysis (diffraction of back - scattered electrons) on a system with electronic and focused ion beam Quanta 200 3D. Studies of the phase composition and crystalline structure of steel samples was performed by X-ray structural analysis on the diffractometer D8 ADVANCE in CuK_α - radiation, using a step with size of 0.02° and a step time of 0.2 s. To reveal the grain edges and carbide phases particles, the chemical etching of thin sections in 4% alcoholic solution of nitric acid was applied (etching time - 5-7 s.). Microhardness of the samples was measured by the diamond indenter indentation method on the device PMT-3M, at the load of 100 g and under a load of exposure - 10 seconds.

Results and discussion

The figure 2 shows the SEM images of the R6M5 steel surface in the initial state, i.e., after a standard heat treatment. It can be seen that the microstructure of R6M5 steel consists of martensite and special carbides (Figure 2a). Carbide particles are uniformly distributed in the matrix and are close to the true spherical shape. From the SEM images, obtained with a backscatter detector, we can see two types of carbides: very bright colors carbides containing elements of higher atomic number and gray carbides containing elements of below-atomic-number (Figure 2b). It is clearly shown that the main phase is martensite, and rounded carbide particles occupy a large part of the volume.

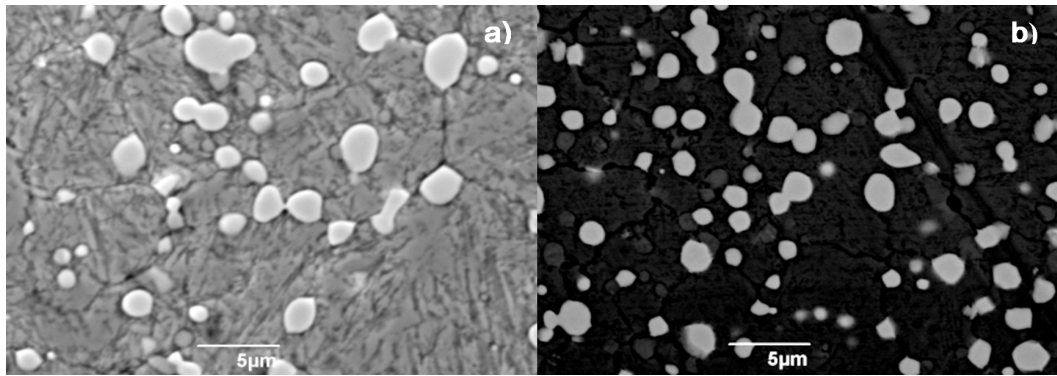


Figure 2. SEM-image of R6M5 steel surface at initial state: a) - image by secondary electrons, b) - image by back scattering electrons.

Figure 3 shows the X-ray diffractograms of R6M5 steel before and after nitriding. X-ray structural analysis showed that in the initial state, i.e., after standard heat treatment, the R6M5 steel structure has martensite (α - phase) and carbides M_6C , MC . After nitriding process, there is a broadening, decrease of intensity and shift toward smaller Bragg angles of the diffraction line (110) of α - phase, indicating the formation of a solid solution of nitrogen in iron, i.e. zone of internal nitriding [11]. The diffraction patterns of the steel R6M5 samples, nitrided at $T = 500^\circ\text{C}$ and $T = 550^\circ\text{C}$, detected interference lines of Fe_4N - phase.

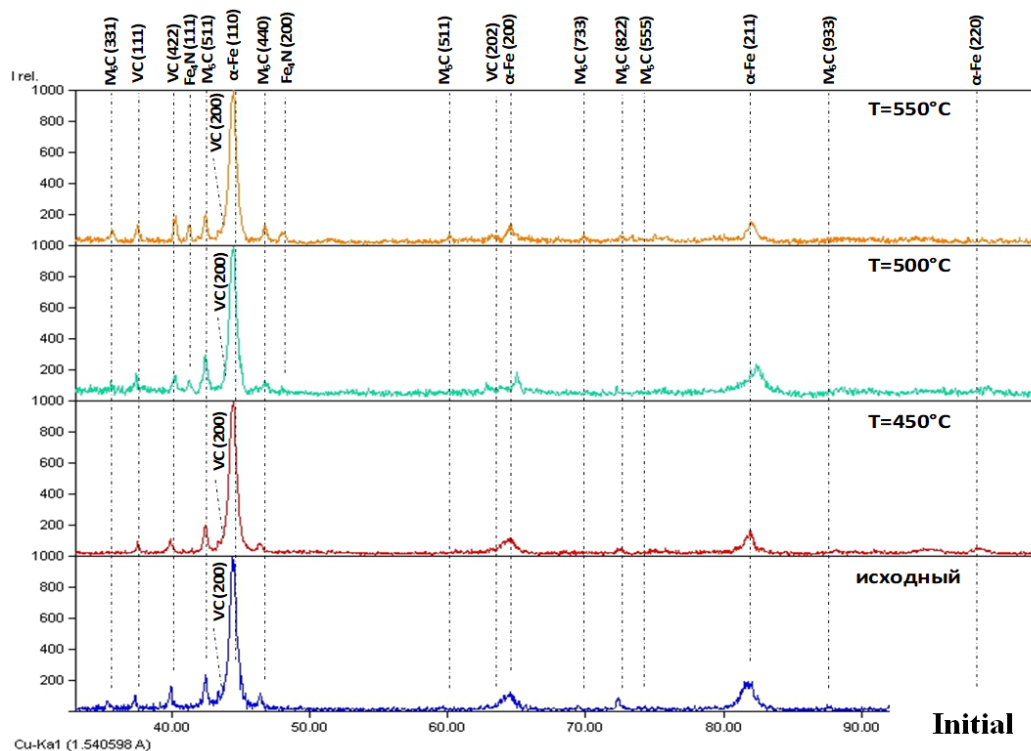


Figure 3. X-ray diffractograms of R6M5 steel before and after nitriding.

As it is well known [12], the main alloying elements of the high-speed steel R6M5 are tungsten, molybdenum, vanadium and chromium. These elements form in steel special carbides: M_6C - based on tungsten and molybdenum, the MC - based on vanadium and $M_{23}C_6$ - based on chromium. X-ray structural analysis results show that the steel structure contains carbides M_6C and MC ,

but carbides $M_{23}C_6$ are absent (Figure 3). In some studies [13,14] there are some indications that after standard heat treatment, steel R6M5 structure contains only carbide M_6C particles. Apparently, this is due to the small volume fraction of MC carbides and similarity of these particles with the matrix, which can not detect them. Also methods, used in these studies, have limitations when detecting carbide particles with a low concentration. Therefore, this study to identify the carbide phases was performed, using special method of electron microscopy - EBSD-analysis.

The suggested configuration of M_6C carbide is between formulas $Fe_3(W,Mo)_3C$ – $Fe_4(W, Mo)_2C$ [15]. In other words, besides molybdenum and tungsten atoms in M_6C carbides, there can be up to 2/3 of the iron atoms from total metal atoms. In addition, the atoms of chromium and vanadium can be dissolved, replacing iron atoms.

Figure 4 shows the results of EBSD-analysis. The EBSD-analysis showed that the M_6C carbides are optimally combined with the cubic phase Fe_3W_3C , and MC type carbide corresponds to the VC phase. However, it is worth noting, that in this case Fe_3W_3C can also mean that other carbide-forming elements are present in the form of M_6C carbides.

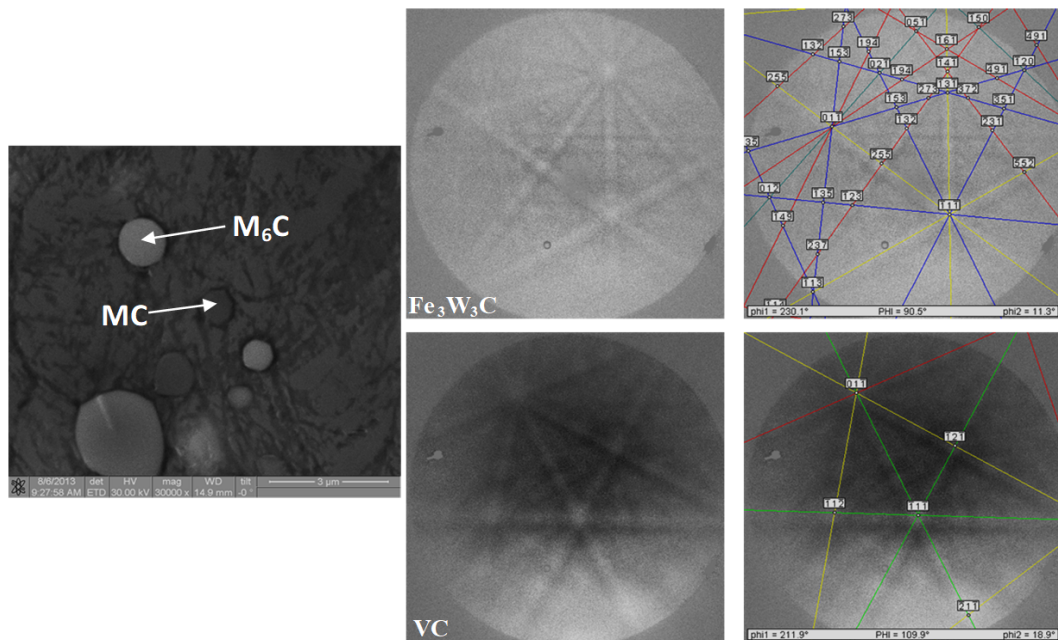


Figure 4. The results of EBSD-analyses of R6M5 steel.

SEM-results showed that after electrolytic-plasma nitriding at $T = 550\text{ }^{\circ}\text{C}$, the fine particles with an average size of $\sim 0.1\text{ }\mu\text{m}$ are formed on the surface of R6M5 steel (Figure 5). And when nitriding at $T = 450\text{ }^{\circ}\text{C}$ and $T = 500\text{ }^{\circ}\text{C}$ such fine particles are not observed. It is assumed that these inclusions are finely dispersed nitrides of alloying elements, in particular chromium [16]. Formation of these finely dispersed nitrides of alloying elements at the temperature of $550\text{ }^{\circ}\text{C}$ is due to the fact that this temperature corresponds to the steel tempering temperature. Tempering steel R6M5 at $T = (550-560)\text{ }^{\circ}\text{C}$ can lead to a dispersion hardening, resulting partial collapse of martensite and separation of fine inclusions of hardening phases [17]. However, X-ray diffraction study revealed no

nitride phases of alloying elements, possibly due to their low concentration and small size. Perhaps this is also due to the formation of fine nitrides, distribution of which over the layer does not provide their detection at the available response level of phase X-ray analysis.

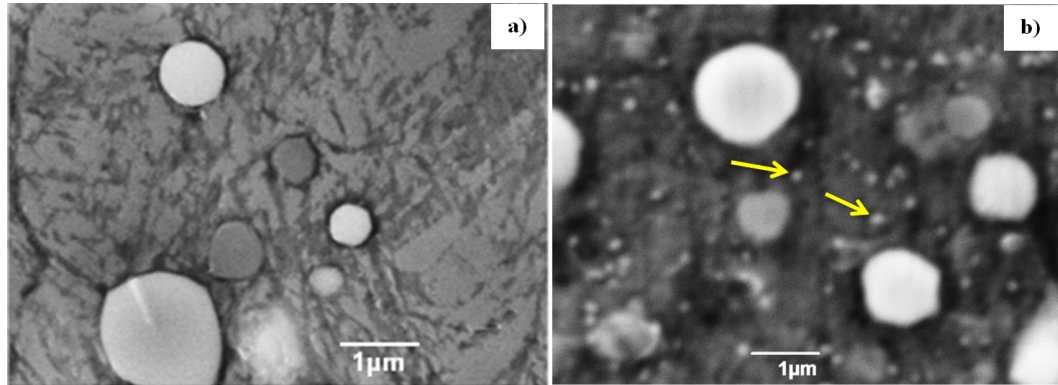


Figure 5. The results of EBSD-analyses of R6M5 steel.

Figure 6 shows the microstructures of the modified surface layer of R6M5 steel samples, nitrided at temperatures of 450°C, 500°C and 550°C for 7 minutes. It can be seen that the dark-etching nitrided layer (representing nitrogen martensite) is observed on the surface. At that, dark-etching zone passes smoothly into the substrate. The thickness of the nitrided layer is on average 25-40 μm and increases with rising nitriding temperature that is quite natural.

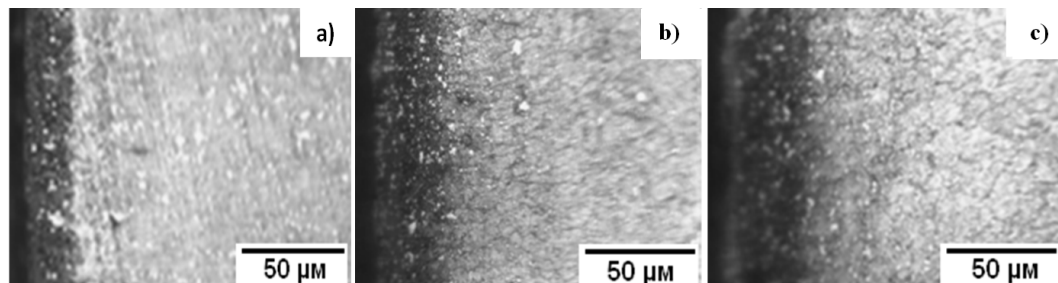


Figure 6. The results of EBSD-analyses of R6M5 steel.

Figure 7 shows the dependence of the microhardness of the R6M5 steel surface layer on nitriding temperature and microhardness distribution on the depth of the nitrided layer. Microhardness of the surface after nitriding increases (1.5-1.6) times. It can be seen a significant increase of microhardness near the surfaces of the treated steel samples. Nature of the transition zone has a smooth transition from the hardened layer to the substrate; herewith the substrate microhardness is not significantly altered. Maximum microhardness depending on the nitriding temperature increases. This is due to the structural-phase state of the surface modified layer. As X-ray diffraction and electron microscopy studies showed, structural and phase states of the R6M5 steel modified layer are varied depending on the nitriding temperature. Thus, when nitriding at $T = 500^{\circ}\text{C}$ and $T = 550^{\circ}\text{C}$, nitride Fe_4N (γ' -phase), along with nitrogen martensite, is formed on the R6M5 steel surface. But at $T = 550^{\circ}\text{C}$, the fine particles with an average size of $\sim 0.1 \mu\text{m}$ are formed on the surface of R6M5 steel.

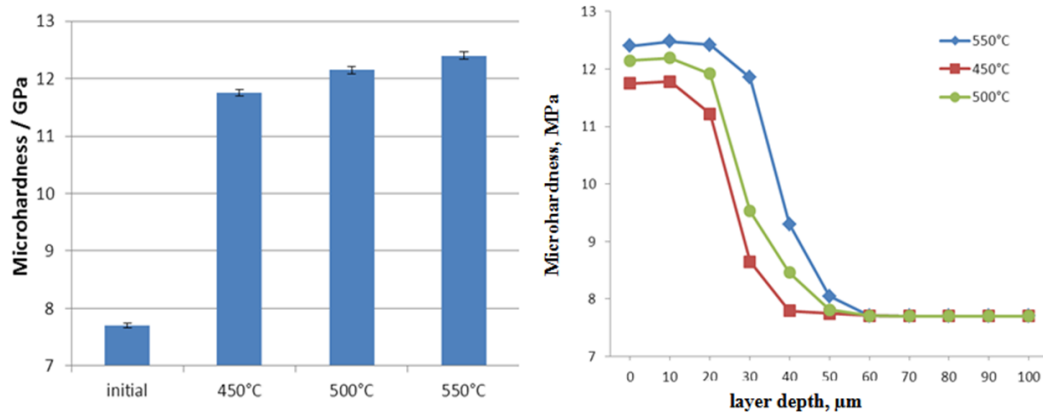


Figure 7. The results of EBSD-analyses of R6M5 steel.

On the basis of X-ray diffraction and electron microscopy studies we can argue that the high hardness of the high-speed steel nitrided layer is associated with the formation of nitrogen martensite and γ' -phase, as well as the separation of the fine inclusions of hardening phases. It should be noted that the formation of the diffusion layer from the nitrogen martensite in the surface layers will have a positive impact on the performance of the cutting tools, made of high-speed steel. This is due to the fact, that the iron nitrides have higher heat capacity as compared with iron [18]. This creates favorable conditions to prevent temperature outbreaks on the surface of the cutting tool.

Conclusion

Analyzing the results obtained in this work, we can assume the following conclusions:

1. Electrolytic-plasma nitriding is an effective way of structure and properties modification, at that at the lower temperatures that allow using it for hardening the heat-treated high-speed steels as a finishing treatment. In addition, changing the process parameters, we can flexibly control the thickness of the modified layer, its phase and structural state, mechanical properties, which are important when developing processes for specific hardenable tools, operated under different conditions;

2. It is determined that steel R6M5 microstructure in the initial state, i.e., after standard heat treatment, consists of tempered martensite and solid carbides M_6C , MC with a diameter of spherical shape. The EBSD-analysis (along with X-ray structural analysis) showed that the M_6C carbides are optimally combined with the phase Fe_3W_3C , and MC type carbide corresponds to the VC phase;

3. It is found that after electrolytic-plasma nitriding of the steel R6M5 surface, a modified layer (representing nitrogen martensite) is formed. Phase composition of the modified layer is changed depending on the nitriding temperature. Thus, when nitriding at $T = 500^\circ C$ and $T = 550^\circ C$, nitride Fe_4N (γ' -phase), along with nitrogen martensite, is formed on the P6M5 steel surface. But at $T=550^\circ C$, the fine particles with an average size of $\sim 0.1 \mu m$ are formed on the surface of R6M5 steel;

4. It is determined that after electrolytic-plasma nitriding of the steel R6M5 surface, the microhardness of its surface layer (depending on the structural-phase state) increases. The main factor influencing the increase of the R6M5 steel microhardness is the formation of nitrogen martensite, Fe_4N (γ' -phase), as well as the formation of fine inclusions of hardening phases in the surface layers.

Acknowledgments

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References

- [1] A.S. Vereshhaka, Rabotosposobnost' rezhushhego instrumenta s iznosostojkimi pokrytijami (Mashinostroenie, Moskva, 1993) 336 s. (In Russian)
- [2] S.N. Grigor'ev, Tehnologicheskie metody povyshenija iznosostojkosti kontaktnyh ploshadok rezhushhego instrumenta (TNT, Staryj Oskol, 2011) 379 s. (In Russian)
- [3] A. da Silva Rocha et al., Surface and coatings technology **165** (2003) 176.
- [4] U. Ion-Dragos et al., Materials testing **55**(1) (2013) 47.
- [5] S.A. Gerasimov et al., Struktura i iznosostojkost' azotirovannyh stalej (Izd-vo MGTU im.N.Je.Baumana, Moskva, 2002) 48 s. (In Russian)
- [6] M. Karimi Zarchi et al., Journal of Materials Research and Technology **2**(3) (2012) 213.
- [7] P. Gupta et al., Surf. & Coat. Technol. **25** (2007) 87.
- [8] I.V. Suminov i dr., Mir materialov i tehnologij. Tom 1 (Izd. Tehnosfera, Moskva, 2011) 464 s. (In Russian)
- [9] M.I. Gol'dshtejn, Grachev S.V., Veksler Ju.G. Special'nye stali (Metallurgija, Moskva, 1985) 408 s. (In Russian)
- [10] M.K. Skakov et al., Stanochnyj park **6**(105) (2013) 30. (In Russian)
- [11] B.N. Arzamasov i dr., Ionnajahimiko-termicheskaja obrabotka splavov (Izd-vo MGTU im. N.Je. Baumana, Moskva, 1999) 400 s. (In Russian)
- [12] Ju.A. Geller, Instrumental'nye stali (Metallurgija, Moskva, 1983) 527 s. (In Russian)
- [13] R. Guenzel et al., Pulsed electron-beam treatment of high-speed steel current tools: structure-phase transformation and wear resistance: 1st International Congress on Radiation Physics, high current electronics and modification of materials **3** (2000) 3037.
- [14] Ivanov Yu. et al., Surface and Coatings Technology **150** (2002) 188.
- [15] H.Dzh. Gol'dshmidt, Splavy vnedrenija. Tom 1 (Mir, Moskva, 1971) 424s.
- [16] S.A. Gerasimov et al., MiTOM **1** (2004) 13. (In Russian)
- [17] I. Artinger, Instrumental'nye stali i ih termicheskaja obrabotka (Moskva, 1982) 312 s. (In Russian)
- [18] K.B. Usmanov, G.I. Yakunin, Vliyanie vneshnikh sred na iznosistojkost' rezhushhikh instrumentov (FanUzSSR, Tashkent, 1984) 160 s. (In Russian)