

Structural and Phase Transformations in the High-Speed Steel as a Result of Electrolytic-Plasma Nitriding

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Abstract— The article investigates the influence of electrolytic-plasma nitriding on structure-phase state of the R6M5 high-speed steel surface layer. It is found that after electrolytic-plasma nitriding on the R6M5 steel surface, diffusion layer is formed, which is a nitrogen marten site. It was showed phase composition of diffusion layer is changing depending on the nitriding. It is found that electrolytic-plasma nitriding lead to accelerated formation of the modified layer. It is determined that after electrolytic-plasma nitriding on the high-speed steel surface, modified layer is formed, consisting only of the diffusion layer. It is determined the influence of the saturation temperature on the diffusion layer structural and phase states.

Index Terms—structure, phase composition, plasma nitriding, high-speed steel, nitrides.

I. 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 nitride layer with a given structure [3-5] on the surface of the work pieces. One of the promising methods for plasma nitriding, which allows significantly reducing the overall processing time, as well as significantly increasing 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-phase states and micro harness of the R6M5 steel surface layer after electrolytic-plasma nitriding.

II. 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. Work piece samples for research have been cut from bars of R6M5 steel in the form of parallelepipeds with 10x30x30 mm³ dimensions. The samples were then subjected to standard for this steel thermal processing: quenching up to 1230°C in oil, and subsequent tempering (three times) at 560°C (the period of each tempering is 1 hour; cooling in air) [9]. After thermal processing, samples have been grounded and polished, and then were subjected to electrolytic-plasma nitriding. Electrolytic-plasma nitriding of the samples was performed on the experimental industrial installation [10], having the following main parts: an electrolytic cell, power supply, automatic control system, cooling system of the electrolyte, the electrolyte supply system (Fig. 1). The electrolytic-plasma nitriding is consisted in the processing of 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 of 20 % carbamide and 10 % 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% carbamide and 10% 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 V, and when exposed at the $T = 450-550^{\circ}\text{C} - 200\text{V}$, nitriding time - 7 minutes. Nitriding temperature was chosen below the tempering temperature,

which ensures the hardness of the matrix at baseline.

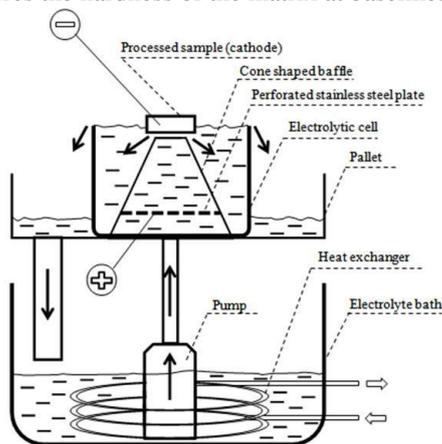


Fig.1. the scheme of electrolyte-plasma nitriding equipment

Metallographic examination was carried out on the optical microscope «ALTAMI-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 phase's particles, the chemical etching of thin sections in 4% alcoholic solution of nitric acid was applied (etching time - 5-7 s.). Micro hardness 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.

II. RESULTS AND DISCUSSION

Figure 2 shows the microstructure of the modified surface layer of R6M5 steel samples, nitrated at temperatures of 450°C, 500°C and 550°C in the period of 7 minutes. It is seen that the surface is observed dark-etching nitride layer consisting from diffusion layer. The dark-etching area is blend to basis. The thickness of the nitride layer is on average 25-40 μm and increases with increasing nitriding temperature, which is completely regular. 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 thermal processing, 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, nitride at $T = 500^\circ\text{C}$ and $T = 550^\circ\text{C}$, detected interference lines of Fe_4N -phase. SEM-results showed that after electrolytic-plasma nitriding 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 4). And when nitriding at $T = 450^\circ\text{C}$ and $T = 500^\circ\text{C}$ such fine particles are not observed. It is assumed that these inclusions are finely dispersed nitrides of alloying elements, in particular chromium [12]. Formation of these finely dispersed nitrides of alloying elements at the temperature of 550°C is due to the fact that this temperature corresponds to the steel tempering temperature. Tempering steel R6M5 at $T = 550\text{-}560^\circ\text{C}$ can lead to a dispersion hardening, resulting partial collapse of martensite and separation of fine inclusions of hardening phases [13]. 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. In order to identify the crystal structure of the second phases, which is formed during nitriding; the fine structure of the R6M5 steel samples before and after nitriding at 550°C for 7 minutes is investigated, using transmission electron microscopy. Fine structure of the R6M5 steel was observed by electron microscopy (Figure 5). Clearly shows that the main phase is the α -phase (martensite) and rounded carbide particles that occupy a large part of the volume. Morphologically carbide phases more clearly faceted. Martensite structure after nitriding more fragmented.

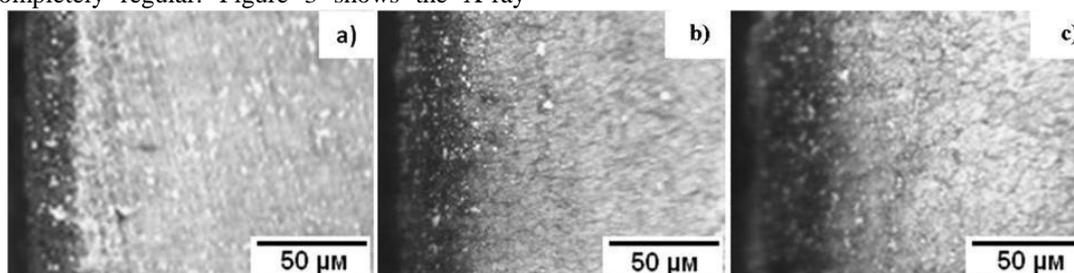


Fig 2. Microstructure of R6M5 steel diffusion layer after nitriding a) $T=450^\circ\text{C}$, b) $T=500^\circ\text{C}$ and b) $T=550^\circ\text{C}$

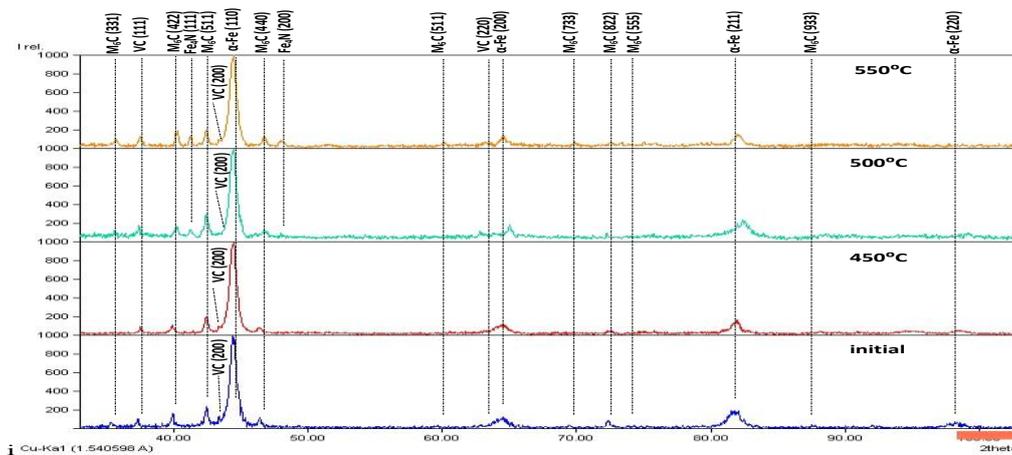


Fig 3. X-ray diffractograms of R6M5 steel before and after nitriding

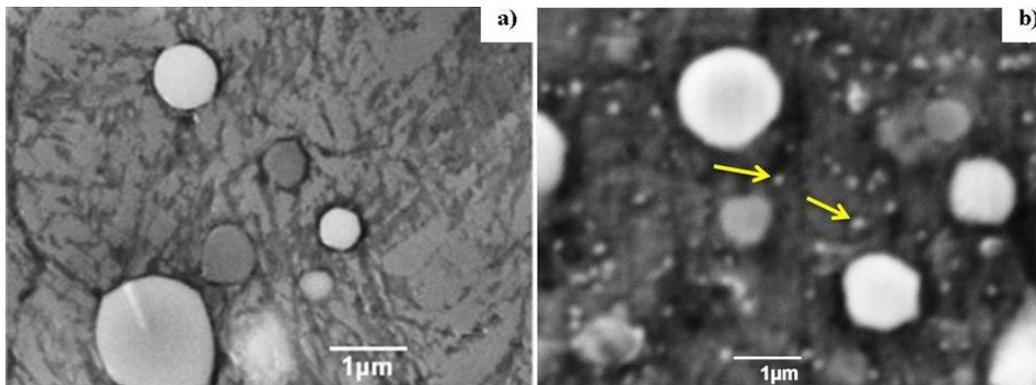


Fig 4. The SEM-image of R6M5 high-speed steel surface after electrolytic-plasma nitriding

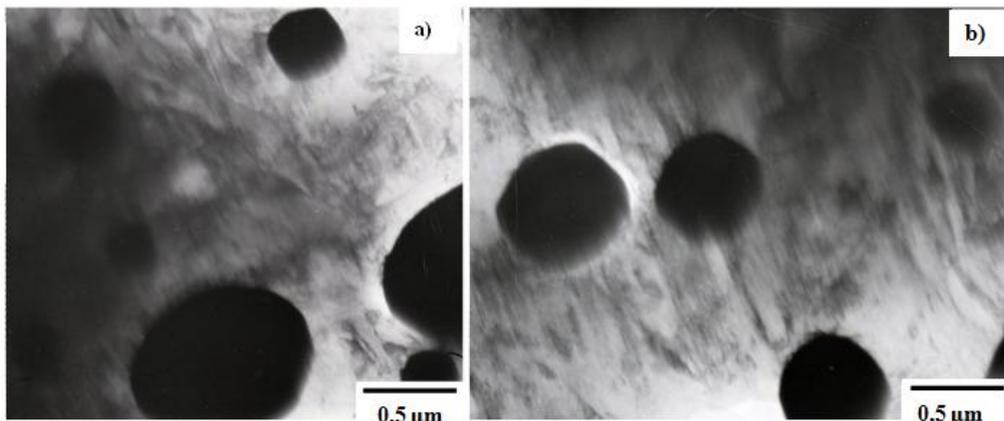


Fig 5. Electron microscope image of the R6M5 steel fine structure before (a) and after nitriding (b)

Electron microscopic images of the R6M5 steel fine structure after nitriding and micro diffraction patterns obtained from carbide, matrix and fine carbides and their indexing schemes are shown in Figure 6. Micro diffraction patterns obtained from matrix are showed that in the R6M5

steel structure after nitriding, Fe_4N nitride (γ' -phase) is presenting (Figure 6 b). Electron-microscopic method confirmed the formation of finely dispersed nitrides in the surface layer after nitriding. Micro diffraction pattern obtained from land with finely dispersed nitrides showed that these nitrides phase corresponds to CrN (Figure 6 c).

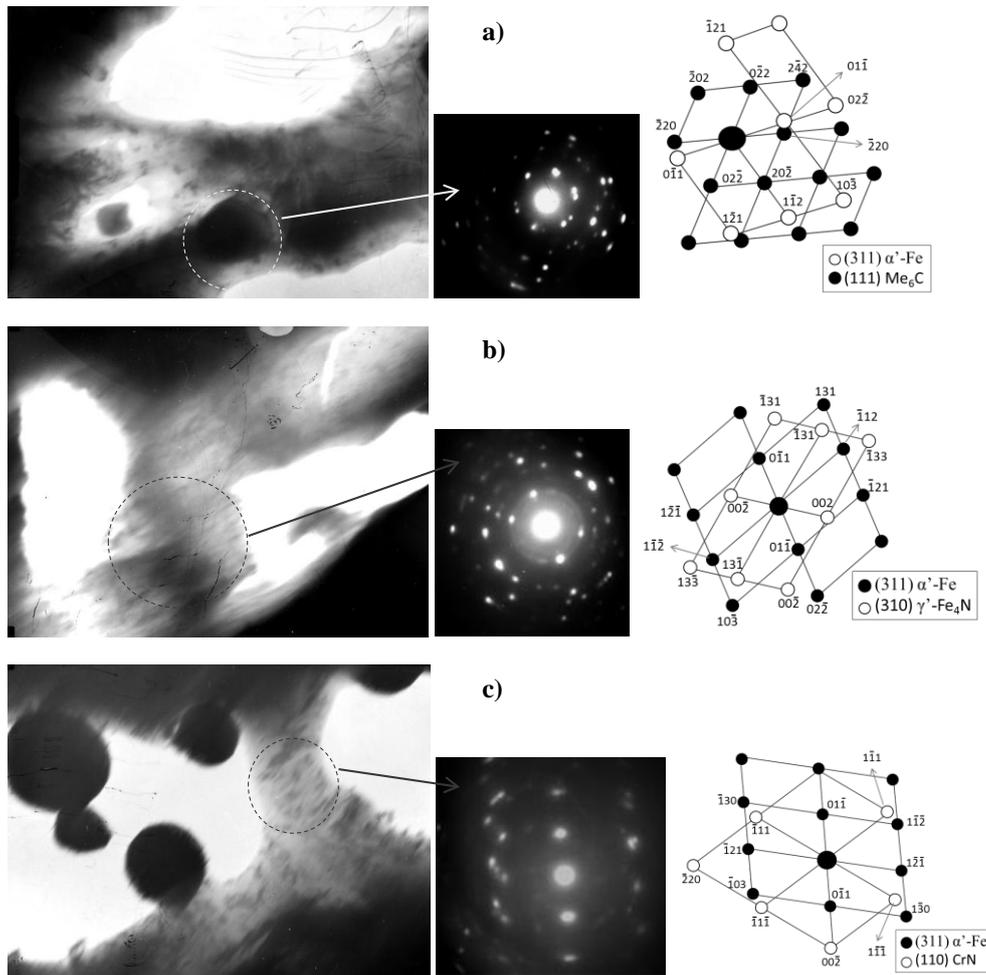


Fig 6. TEM image of the R6M5 steel fine structure after nitriding and microdiffraction image obtained from the carbide (a), the matrix (b) and fine carbides (c) and their indexing scheme

Thus, based on electron-microscopic researches it is determined that after nitriding on the surface layer, fine nitrides are formed. Fine nitrides are small irregularly shaped particles (Figure 7 b, indicated by arrows). Micro diffraction analysis showed that the crystal structure of these nitrides corresponds to CrN nitride (cubic lattice, space group Fm3m).

Micro diffraction analysis also showed that after nitriding on the surface layer presents Fe₄N nitrides (γ' -phase), platelet-shaped (Figure 7 a, indicated by arrows).

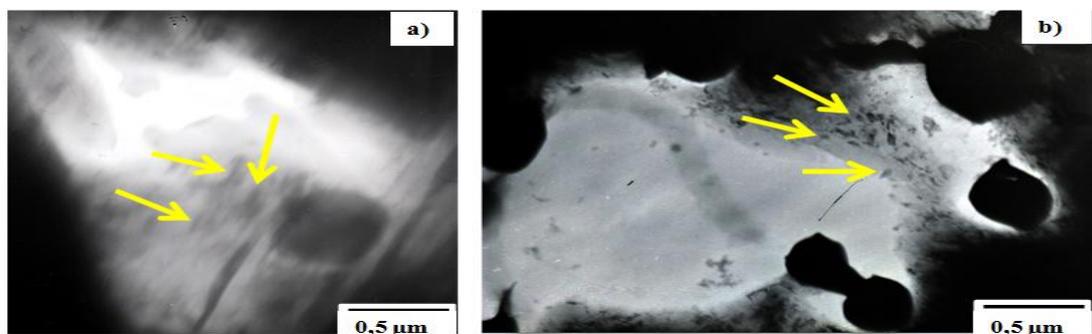


Fig 7. Electron microscopic images of the R6M5 steel fine structure after nitriding

Thus, we studied the structural and phase transformations in the surface layers of the R6M5 high-speed steel after electrolytic-plasma nitriding. It is assumed that the structural changes entail higher strength material of high-speed steel.

III. CONCLUSION

Analyzing the results obtained in this work, the authors conclude:

1. It is established that electrolytic-plasma nitriding lead

accelerated the formation of the modified layer. In addition, by changing the process parameters the thickness of the modified layer can be flexibly controlled, its phase and structural state, which is important when developing processes for specific types strengthened by instruments operated under different conditions.

2. It is established that after electrolytic-plasma nitriding on the high-speed steels surface modified layer is formed, consisting only of the diffusion layer.

3. Influence of saturation temperature on the structural and phase states of the diffusion layer. In the diffusion layer consisting of nitrogen martensite with increasing nitriding temperature from 450°C to 500°C - a redundant γ' -phase and at a temperature 550°C nitriding, MN-type fine nitrides is formed.

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