# Mazhyn SKAKOV<sup>\*</sup>, Bauyrzhan RAKHADILOV<sup>\*</sup>, Michael SHEFFLER<sup>\*\*</sup>

# CHANGE OF STRUCTURE AND WEAR RESISTANCE OF P6M5 STEEL FROM PROCESSING IN ELECTROLYTE PLASMA

# ZMIANA STRUKTURY I ODPORNOŚCI NA ZUŻYWANIE STALI P6M5 W WYNIKU OBRÓBKI ELEKTROLITYCZNO-PLAZMOWEJ

## Key words:

formatting; modification, electrolytic plasma nitriding, carbonitriding, wear resistance

#### Słowa kluczowe:

formowanie; modyfikowanie, azotowanie elektrolityczno-plazmowe, cyjanowanie, odporność na zużywanie

#### Summary

Mechanical characteristics of nitrated and carbonitriding in electrolyte plasma P6M5 steel surface layers are investigated in the research. Perspective technology of cutting tool electrolyte-plasma treatment is displayed.

<sup>&</sup>lt;sup>\*</sup>D. Serikbaev East Kazakhstan State Technical University, Ust-Kamenogorsk, Kazakhstan.

<sup>\*\*</sup> University of Otto-fon-Guericke, Magdeburg, Germany.

Comparative research of structure, phase composition of fast-cutting P6M5 steel modified surface layers after electrolyte plasma treatment was carried out by scanning-electron and light microscopy, and X-ray structure analysis methods.

### **INTRODUCTION**

It is well known that cutting tools should be durable, strong, have high heat resistance and corrosion resistance. These properties can be achieved by applying surface chemical treatments and by thermal processing of metals. The most appropriate methods are the processes of carburizing, boriding, nitriding, carbonitriding, and sulfiding [L. 1, 2]. The general major drawback of these processes is that they are very time consuming. One of the major challenges in chemical-thermal treatment is the significant intensification of diffusion saturation processes by processing methods of concentrated flows of energy [L. 3, 4]. Among them, the most promising energy-saving method is the method of chemical-thermal treatment in electrolytic plasma [L. 5]. It was found that the rate of the diffusion of elements in the surface layer during the processing in the electrolyte plasma is significantly higher than in ordinary processes [L. 6]. When processing in plasma electrolyte, there are certain changes in the structural-phase state and material properties in thin surface layers due to the physical effects of high temperature plasma ions and electrical discharge. Therefore, the research of peculiar structure change, properties and mechanisms of hardening phase steel P6M5 extraction after chemical and thermal processing in the electrolyte plasma is of great scientific and practical interest in terms of clarifying the general laws of structural phase transitions in steels and the development of new progressive ways of processing materials in order to improve their practical important properties.

In connection with the above, the objective of this research paper is the investigation of the mechanisms that change structure, phase composition, microhardness, and wear resistance of steel P6M5 during nitriding and carbonitriding in electrolytic plasma.

#### MATERIAL AND METHODS OF RESEARCH

In accordance with the objective fast-cutting tungsten-molybdenum steel P6M5 (0.80 - 0.88 C, 3.8 - 4.4 Cr, 5.5 - 6.5 W, 1.7 - 2.1 V, 5.0 - 5.5 Mo) was selected as an object of study [L. 7]. The choice of material for research is based on the fact that steel P6M5 is the most common in the metalworking, because it is a typical, fast-cutting steel with moderate heat resistance.

Samples for research in the form of parallelepipeds with dimensions 10h30h30 mm<sup>3</sup> were cut from steel P6M5 bars in "as-received" condition.

Before electrolytic-plasma processing, blanks were subjected to common heat treatment for this type of steel: quenching from 1230°C in the oil and the subsequent triple annealing at 560°C (duration of each annealing is 1 hour, cooling in air) [L. 8]. Then the samples were polished on all sides to 1 mm depth. Before the cementation, the surfaces of the samples were carefully degreased.

Electrolyte-plasma processing was done by nitriding and nitrocarburizing of nitrogen and carbon-aqueous solution in the cathode mode (**Fig. 1**). One powerful rectifier served as a source of power, which gives the maximum output of 360 V/60 A in the form of direct current. The samples were processed by heating them to a temperature of  $750-900^{\circ}$ C, and then maintaining these temperatures using the electric potential in the layer of plasma created between the liquid electrode (electrolyte) and the surface of the cathode (sample). Processing conditions are presented in **Table 1**.

Table 1.	Technical	paramete	rs of ele	ctrolyt	ic-plasma	processing
Tabela 1	. Parametry	obróbki e	lektrolity	yczno-j	olazmowej	

Types of processing	Composition of electrolyte (%, mass)	Curing temperature T,°C	U, V	I, A	t, min
nitriding	Top of Form Ammonium fluoride (15%) + sodium carbonate (10%) and glycerine (5%) + water	550, 650, 750	110	30	5
carbonitriding	Carbamide (15) + sodium carbonate (10) and glycerine (5) + water	550, 650, 750	175	30	5

Research on the phase composition and crystalline structure of the samples were performed by X-ray analysis on a diffractometer X'Pert Pro in CuK  $\alpha$  – radiation. Surface morphology was studied on an optical microscope «NEOPHOT-21", and scanning electron microscope JSM-6390LV, equipped with energy dispersive analysis installation. The microhardness of the surface layers of the samples before and after treatment was measured by diamond indenter pressing in the PMT-3 with load of 100 g and holding load of 10 s. The wear investigation was conducted on a instrument for testing the abrasion of the samples produced by not rigidly fixed abrasive particles (GOST 23.208-79). Abrasive wear resistance of tested materials was evaluated by comparing its wear with the wear of a reference sample (steel 45).

# **RESULTS AND THEIR DISCUSSION**

In structure study of steel P6M5 samples, phase and structural changes were detected in the surface layers subjected to chemical and thermal processing in the electrolytic plasma. **Figure 1** shows the microstructure changes of the steel surface layer after nitriding and carbonitriding for 5 min. We observed an increase in carbides, a depletion of ferrite by alloying elements, and an enrichment of carbon. As a result, large number of dispersed nitrides and carbides from the alloying components are extracted from the solid solution, i.e. disperse hardening occurs. The microstructure of the hardened surface of the processed sample is of a fine-grained martensitic structure with dispersed insoluble inclusions of carbides and nitrides.



- Fig. 1. The microstructure of the surface of steel P6M5: a before and after carbonitriding at temperatures of b 650°C, c 750°C, nitriding at temperatures of d 550°C, e 650°C, f 750°C
- Rys. 1. Mikrostruktura powierzchni stali P6M5: a przed i po cyjanowaniu w temperaturze, b – 650°C, c – 750°C, azotowaniu w temperaturze d – 550°C, e – 650°C, f – 750°C

X-ray structure research was done to define surface phase composition. Reflections of  $\varepsilon$ -phase Fe<sub>2</sub>N and Fe<sub>3</sub>N were detected (**Figure 2**) on the surface of the sample of steel P6M5 after nitriding and plasma electrolyte carbonitriding at 550...750°C. It is known [L. 12] that iron nitrides have higher heat capacity than iron, which is useful in creating tool surfaces that are resistant to damage from high temperatures. It should be noted that, on diffraction patterns taken from the samples nitrided at 750°C, a weak peak appears, corresponding to  $\gamma$ -phase (nitrogen austenite).





Fig. 2. Fragments of diffraction patterns of steel P6M5: a – before and after nitriding at temperatures of b – 550°C, c – 650°C, d – 750°C

Rys. 2. Fragmenty dyfragrometru stali P6M5: a – przed i po azotowaniu w temperaturze, b – 550°C, c – 650°C, d – 750°C

**Figure 3** shows the even distribution of the microstructure of the diffusion layer of steel P6M5 after nitriding. Analysis of microstructure of sample P6M5 steel after processing showed the presence of two zones:

The first zone is a continuous layer of nitrides of iron and alloying elements.

The second zone, the diffusion region, is a nitrogen ferrite with carbide and fine-dispersed nitride inclusions.



- Fig. 3. The microstructure of the diffusion layer of steel P6M5 after nitrating at temperatures as 750°C
- Rys. 3. Mikrostruktura warstwy dyfuzyjnej stalil P6M5 po azotowaniu w temperaturze 750°C

Figure 4 shows dependence between the processing temperature and the thickness and micro-hardness of the nitrated and nitro-cemented layer. The graphs show that the saturation rate micro-hardness increases with increasing treatment temperature. Notice that there is an increase in the depth of the modified layer of approximately 25  $\mu$ m at a temperature of 550°C and approximately 40  $\mu$ m at a temperature of 750°C. The obtained results are in good agreement with the well-known published data [L. 10]. It should be noted that, in a relatively short time (5 min.) in the investigated temperature range of 550-750°C, the nitrated layer is formed with a sufficiently large thickness.



Fig. 4. Dependence of the thickness and micro-hardness of the nitrated and nitro-cemented layer of steel P6M5 from temperature

The increase in micro-hardness is mainly due to the formation of a solid solution of nitrogen in iron. The micro-hardness of all modified layers in the electrolyte plasma is sufficiently high (H $\mu$  = 9.5 – 13.5 GPa). The high hardness of the modified layer is due to the fact that, apparently, it consists of nitrogen martensite and dispersed nitrides of the  $\varepsilon$ -phase and  $\gamma$ -phase and nitrides of alloying ingredients Mo, W, Cr, V [L. 11]. However,  $\gamma'$ -phase and nitrides of molybdenum, vanadium and chromium are not detected by X-ray analysis, possibly due to their dispersion and a small quantity.

**Figure 5** shows the results of the test on durability. It is seen that wear resistance after nitriding and carbonitriding increases. The high value of wear resistance is achieved at the temperature of 650°C. Further increase in temperature of up to 750°C leads to a decrease in wear resistance. This is due to the formation of an austenitic phase in the steel structure, as shown by the results of x-ray analysis at 750°C where diffraction appears of the reflex  $\gamma$ -Fephase (**Figure 2d**). As is well known [**L. 12**], the austenite is an unwanted structural component in tool steels that reduces the wear resistance and thermal conductivity.

Rys. 4. Zależność grubości i mikrotwardości warstwy azotowanej i cyjanowanej stali P6M5 od temperatury



- Fig. 5. Wear resistance of steel P6M5 depending on nitriding and nitro-cementation temperature
- Rys. 5. Odporność na zużywanie stali P6M5 w zależności od temperatury azotowania i cyjanowania

# CONCLUSIONS

Analysing the research results obtained, we can draw the following conclusions:

- 1. A means of electrolytic plasma nitriding in cathodic mode has been described that produces surface modification of fast-cutting steels and the high kinetic efficiency of the diffusion saturation process.
- 2. It has been experimentally demonstrated that a solid electrolyte modified layer consisting of particles of  $\epsilon$ -phase Fe<sub>2</sub>N and Fe<sub>3</sub>N is formed on the sample surface of steel P6M5 after carbonitriding and nitriding in electrolyte plasma.
- 3. It has been established that a significant increase in the microhardness in the surface layers of steel P6M5 (1.3 1.8 times) is a result of nitriding and nitrocarburizing in electrolyte plasma.
- 4. It was found that nitriding in electrolytic plasma results in a 60% increase in wear resistance at a temperature of  $650^{\circ}$ C. Increasing the saturation temperature up to the temperature of  $750^{\circ}$ C leads to a reduction in wear resistance. This is due to the fact that nitriding at a temperature of  $750^{\circ}$ C leads to the emergence of the austenitic  $\gamma$ -Fe-phase in the structure of the steel, which has a negative effect on the wear resistance of steel P6M5.
- 5. The thickness and microhardness of the surface layer created by high-speed steel nitriding in plasma electrolyte is greatly influenced by the composition of nitrogen-containing electrolyte and time and temperature.

#### ACKNOWLEDGMENT

The present research work was done on the basis of the Cooperation Agreement between D. Serikbaev East Kazakhstan State Technical University, Tomsk State Architecture and Building University, Tomsk, Russia, 2008 and University of Otto von Guericke, Germany, 2008, in accordance with the contract of Joint-Stock Company "Science Fund of the Republic Kazakhstan" on the theme "Development and implementation of innovative technologies of electrolytic-plasma hardening of drilling tools material."

#### REFERENCES

- 1. Beliy A.V., Karpenko G.D., Mishkin N.K., Struktura i metodi formirovania iznosostoikih poverhnostnih sloev. M.: Mashinostroenie, 1991. p.208
- Bayati M., Molei R. Zhanhorban K., Poverhnostnoe legirovanie uglerodistih stalei iz elektricheskoi plazmi // Metallovedenie I termicheskaya obrabotka metallov, 2011, № 2 (668), pp. 42–45.
- Zabelin S.F., Ob aktivatsi I kineticheskoi teorii protsessov diffuzionnogo nasishenia metallov pri himiko-termicheskoi obrabotke // Materialovedenie, 2004, № 7, p. 17–22.
- Saraev Y.N. Barns, Shtertser A.A., Orishin A.M., Ilyushenko A.F., Skakov M.K., Kompleksni podhod k povisheniy ekspluatatsionnoi nadiojnosti detalei I izdelii // Tehnologia mashinostroenia, 2011, № 8, p. 39–42.
- Suminov Y.V., Belkin P.N. etc., Mir materialov I tehnologii. V 2-h tomah, Tom 1, M. izd. Tnosfera, 2011, - 464 p.
- Gupta P., Tenhundfeld G., Daigle E.O., Ryabkov D. Electrolytic plasma technology: Science and engineering - an overview / / Surf. & Coat. Technol. In 2007. V. 25. P. 8746.
- 7. Poznyak L.A., Tishaev S.I., Skrynchenko Y.M., Instumentalnie stali: Spravochnik. Moscow: Metallurgiya, 1977, 167 p.
- Kremnev L.S., Vinogradova L.A., Onegin A.K., Sapronov I.J., Osobennosti sostava, strukturi I svoistv bistrorezhushih stalei dlia metallorejushego instumenta s ionno-plazmennimi pokriniami na osnove nitrida titana // Metallovedenie I termicheskaya obrabotka metallov, 2012, № 1 (679), p.4–9.
- Bunin K.P., Vovchan V.I., Pedan L.G., Strukturoobrazovanya pri izotermicheskom nauglerojivanii jeleznih splavov, legirovannih molibdenom I volframom // Izv. AN SSSR. Metalli. – 1975. - №3. – p.164–168.
- 10. Lahtin U.M., Teoria I tehnologia azotirovanya / Lahtin U.M., [i dr.]. M.: Metallurgya, 1991. 320 p.
- Arshinger I, Instrumentalnie stali I ih termicheskaya obrabotka: spravochnik. M.: Metallurgya, 1982. – 313 c.

#### Streszczenie

W przeprowadzonych badaniach określano charakterystykę mechaniczną stali P6M5 azotowanej i cyjanowanej poprzez obróbkę elektrolitycznoplazmową. Przedstawiono obróbkę elektrolityczno-plazmową, która jest przyszłościowa dla obróbki narzędzi obróbkowych. Badania porównawcze struktury, budowy fazowej stali szybkotnącej P6M5 z modyfikowaną warstwą wierzchnią w trakcie obróbki elektrolityczno-plazmowej prowadzono z wykorzystaniem mikroskopii skaningowej i optycznej oraz analizy rentgenowskiej.