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Change in high-temperature wear resistance of high-speed steel by plasma nitriding

The present paper contains results of tribology testing the surface layer of high-speed steel nitrated in electrolytic plasma. Tribology tests were conducted under the temperature of 500 °C, 550 °C and 600 vC degrees using ball-on-disc method. Such parameters as wear intensity and friction coefficient were applied to analyze wear resistance. It is found, that wear resistance of nitrated layer of high-speed steel changes depending on ambient temperature and it showed higher resistance against the wearing process up to 550 °C degrees. It is established, that the rise in testing temperature up to 600 °C degrees resulted in degradation of wear resistance and increase in friction coefficient. The paper addresses the main factors improving wear resistance increase of high-speed steel surface layer after electrolyte-plasma nitriding. The optimum condition of electrolyte-plasma nitriding of high-speed steel P6M5 in electrolyte on the basis of carbamide which allows to make saturation of surface by nitrogen from low-temperature plasma is experimentally positioned and to receive the modified bed of high hardness and a wear resistance. It is positioned that a major factor responsible for a wear resistance of the nitriding layers at high temperatures is formation of the nitride martensite which possesses the major heat capacity in comparison with iron, and finely divided nitride particles.

Keywords: tribology testing, wearing process, adhesion, nitriding, electrolyte-plasma processing, high-speed R6M5 steel.

Cutting tools aggressiveness is mainly determined by the condition of surface layer [1]. Various methods of thermal and chemical-thermal treatment are widely used to improve hardness, strength and wear resistance. There have recently been increasingly frequent development and application of techniques and approaches of plasma nitriding that enable eliminating shortcomings of traditional nitriding methods [2] and much more. Plasma nitriding provides true-to-structure nitrated layer formation on the surface of work piece thereby increasing the wear resistance of the tool and its thermal endurance. Nitrated tool surface with low friction coefficient and well anti-friction properties provides slighter chip removal and prevents its pickup on cutting edges with the formation of wear craters that makes it possible to get cutting speed faster [3-5]. What's more, today engineering is characterized by complicated operation conditions of cutting tools associated with high level of operating voltages, wide temperature range, corrosive environments, etc. Therefore, special requirements shall be met for high wear resistance when cutting tools' operation under high temperatures [6, 7]. In this regard, the present paper investigates wear resistance of nitrated samples of high-speed steel R6M5 under high testing temperature.

In accordance with the purpose, tungsten-molybdenum high-speed steel R6M5 has been chosen as object of study. R6M5 steel has been selected owing to its much widespread use in metal treatment and mild thermal endurance.

The 3D cubed sample workpieces with size of 10x30x30 mm³ were cut out of R6M5 steel bars as received. Then the samples underwent conventional thermal treatment: quenching from 1230 °C in the lubricant followed by three-stage draw at 560°C degrees (duration of each draw is for 1 hour, cooling in the air) [8]. After thermal treatment, the samples were grinded, polished and underwent electrolytic-plasma nitriding in carbamide-based electrolyte.

Electrolytic-plasma nitriding of the samples was performed on a pilot plant [8] equipped with electrolytic cell, power supply, automatic control system, electrolyte cooling system and electrolyte supply system. The treatment was carried out in the electrolyte of aqueous solution containing 20 % of carbamide and 10 % of sodium carbonate as follows: the samples were nitrified at 450, 500 and 550 °C degrees for 7 minutes.

The microhardness of the samples was measured by diamond indenter pressing-in method in PMT-3M machine with a load of 100 gram and aging under load for 10 seconds. Surface roughness was measured by a portable electronic device Diavite DH-5.

Samples' abrasive wear resistance was measured in specialized testing machine designed for checking abrasion by friction on rigidly fixed particles by rotating roller-on-flat surface method in accordance with

GOST 23.208-79 that corresponds to the US Standards ACTM C 6568. For abrasion tests, samples' surfaces were ground and polished to the size of roughness equal to $R_a=1.2 \mu\text{m}$, afterwards they were cleaned with acetone and dried out. Cylindrical rubber roller pressed by radial surface to the flat surface of the sample by force of 22 Newton was rotated at a frequency of 1s^{-1} . Rate of arrival of abrasive particles between the rubber wheel and the sample namely to the test region was 41-42 g/min. Synthetic corundum with grain size of 200...250 microns was used as abrasive particles. Abrasive wear was measured by weighing on analytical balance ADV-200 with accuracy up to 0,0001 gram. In total, abrasive wear was 28.8m long. Before measuring, the samples were blown with compressed air to remove remaining sand.

Tribology tests of R6M5 steel samples were performed in high-temperature tribometer THT-S-BE-0000 under 20 °C, 500 °C, 550 °C and 600 °C degrees by ball-on-disc method in compliance with International Standards ASTM G 99 (Fig. 1) [9]. The wear resistance was characterized by wear intensity and friction coefficient. A ball with diameter of 6.0 mm made of certified Al_2O_3 was used as a counterbody. The tests were carried out by force of 1N, linear speed of 2 cm/sec, radius of wear curvature - 5 mm and friction path - 25 m. The wear resistance was estimated by the wear rate and friction coefficient. The values of wear rate are averaged for each position according to testing results of three samples.

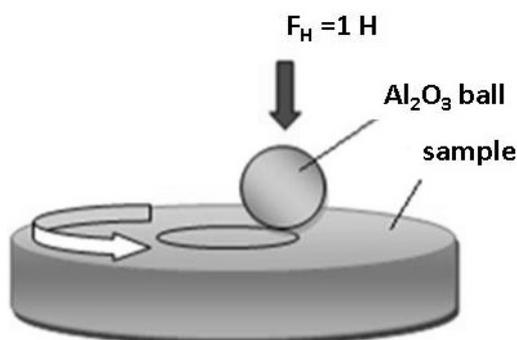
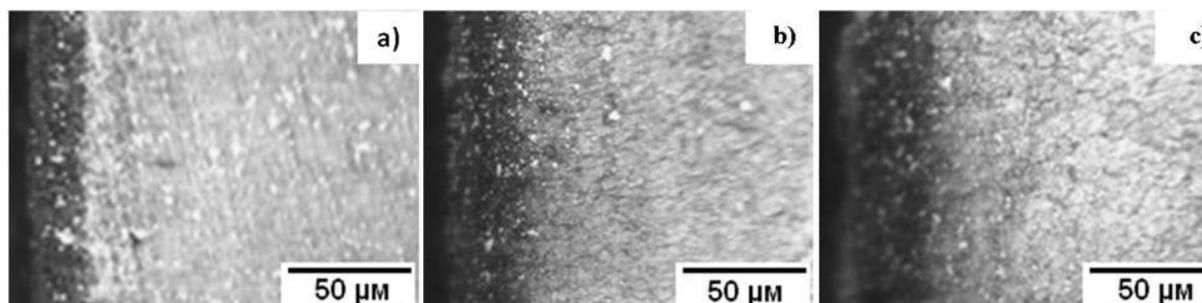


Figure 1. Ball-on-disc tribology test method

Figure 2 illustrates microstructure of modified surface layer of R6M5 steel samples nitrated under 450 °C, 500 °C and 550 °C degrees for 7 minutes. It's clearly seen that there is a dark-etching nitrated layer as nitrous martensite. It is known that R6M5 consists of martensite (α -phase) and solid carbides M_6C and MC in initial state after standard thermal treatment [10]. Dark-etching zone herewith is slipping the base zone. Nitrated layer is about 25-40 μm thick and increases in the wake of nitriding temperature rising.



a – $T = 450 \text{ }^\circ\text{C}$; b – $T = 500 \text{ }^\circ\text{C}$; c – $T = 550 \text{ }^\circ\text{C}$

Figure 2. Microstructure of modified R6M5 surface layers after nitriding

Figure 3 demonstrates changes in wear intensity J , relative wear resistance to abrasive wear \mathcal{E} and microhardness H depending on the nitriding temperature for 7 minutes with no break in the process. It is seen that R6M5 samples nitrated under 550 °C degrees demonstrate the highest microhardness and the lowest wear intensity in comparison with other samples. It is supposedly linked to formation of fine nitrides after nitriding at $T = 550 \text{ }^\circ\text{C}$. It can be concluded that nitriding at $T = 550 \text{ }^\circ\text{C}$ is the best mode of electrolyte-plasma nitriding for R6M5 steel in carbamide-based electrolyte. Therefore initial samples and the samples nitrated at $T = 550 \text{ }^\circ\text{C}$ underwent high-temperature tests.

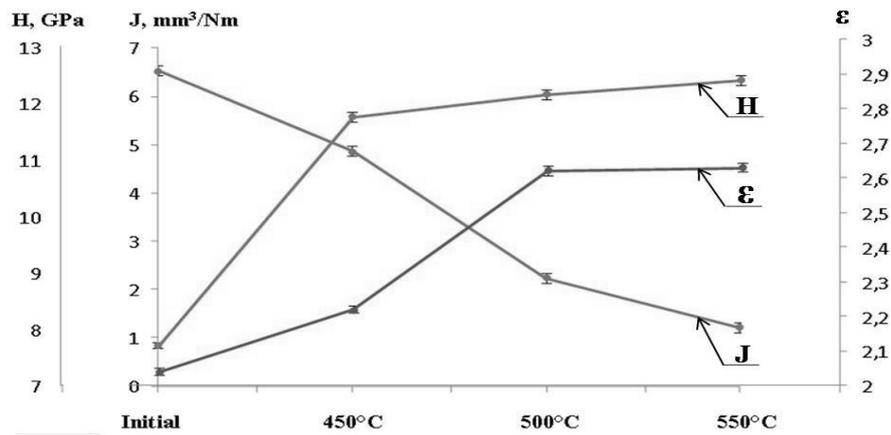
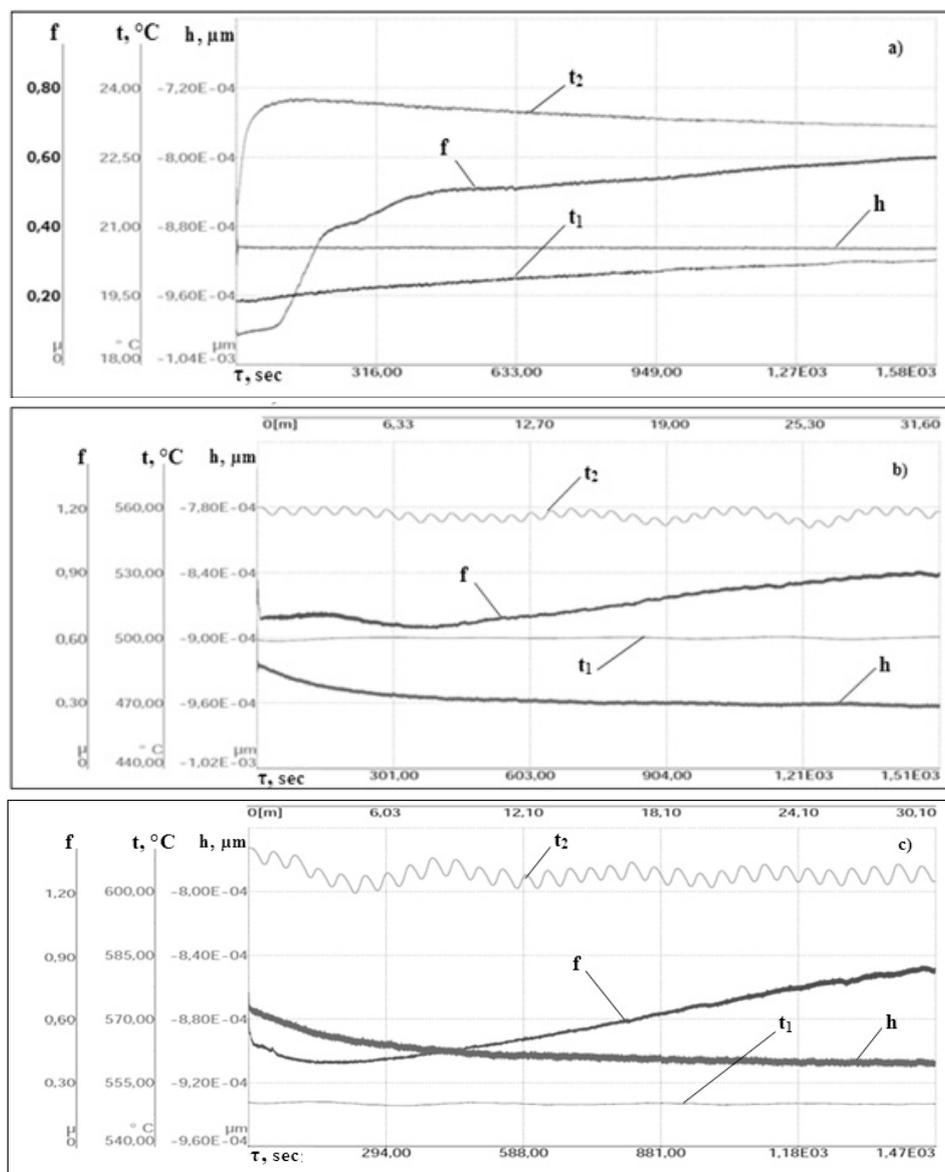


Figure 3. Changes in wear intensity J, relative wear resistance to abrasive wear ϵ and microhardness H depending on the nitriding temperature



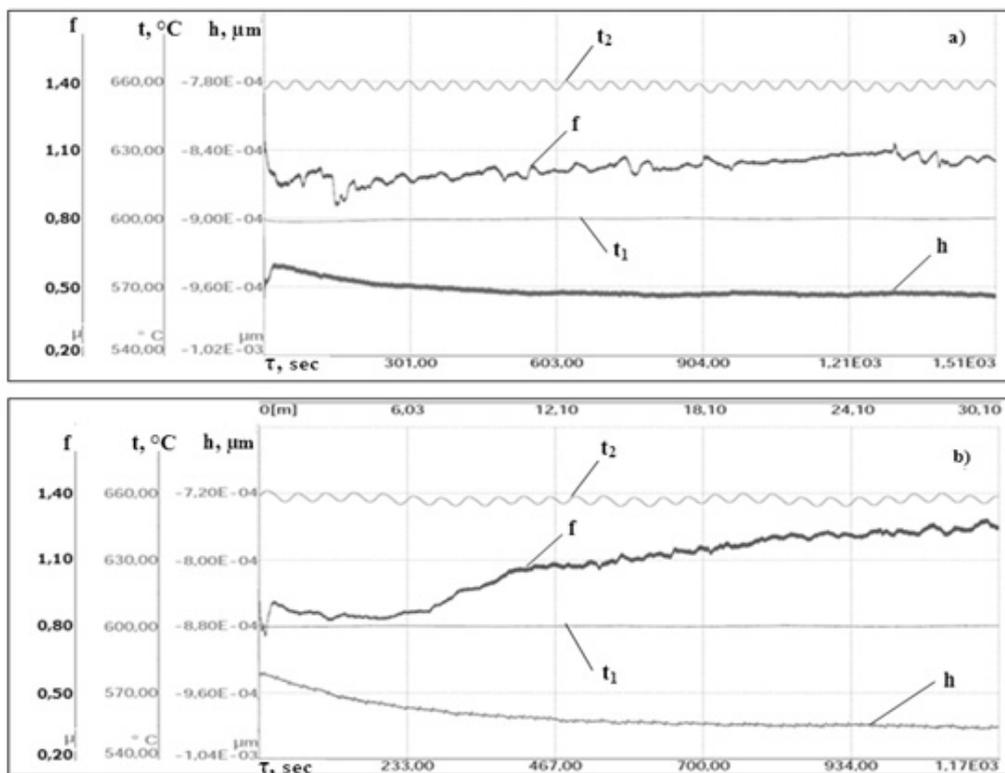
a – at $T=20$ °C; b – at 500 °C; c – at 550 °C temperature

Figure 4. Depending diagrams of friction coefficient f , depth of counter body penetration h , sample temperature t_1 and furnace t_2 on friction time τ when testing of R6M5 nitrated samples

Figures 4, 5 provide results of tribology high-temperature tests of R6M5 samples underwent nitriding at $T = 550^{\circ}\text{C}$.

Figure 4 shows curve changes in friction coefficient, depth counterbody penetration depending on friction time in tests of R6M5 samples nitrated at $T = 20^{\circ}\text{C}$, 500°C and 550°C degrees. There is an increase in friction coefficient under high temperatures. However, changes in testing temperature within $500\text{--}550^{\circ}\text{C}$ degrees hardly affect the friction coefficient.

Figure 5 illustrates curve changes in friction coefficient, depth of counterbody penetration depending on friction time when testing R6M5 samples at $T = 600^{\circ}\text{C}$ before/after nitriding. Rising of testing temperature up to 600°C degrees leads to significant increase in friction coefficient of initial and nitrated samples in comparison with those tested under room temperature, 500 and 550°C degrees. Friction coefficient herewith has larger value in the course of test keeping enough high level.



a – before nitriding; b – after nitriding

Figure 5. Depending diagrams of friction coefficient t , depth of counterbody penetration h , sample temperature t_1 and furnace t_2 on friction time τ when testing of R6M5 nitrated samples at $T=600^{\circ}\text{C}$

Figure 6 shows dependence of wear intensity of initial and nitrated R6M5 steel samples on testing temperature. In tests under room temperatures the wear intensity of nitrated steel samples was lower than of those not nitrated. The wear intensity of nitrated samples in tests at $T = 500^{\circ}\text{C}$ and 550°C is equal about $3,1 \cdot 10^{-4}$ and $3,5 \cdot 10^{-4} \text{ mm}^3/\text{Nm}$; and at $T = 600^{\circ}\text{C}$ it is $6,7 \cdot 10^{-4} \text{ mm}^3/\text{Nm}$. Considerable increase in wear intensity for nitrated and non-nitrated steel samples can be observed at testing temperatures of 500°C and 550°C degrees. Much higher increase in wear intensity is seen under 600°C degrees. Degradation in wear resistance and increase in friction coefficient under testing temperature of 600°C might be due to processes like coagulation or diffusion growth of coherent particles of nitrated phase. According to [10], diffusion processes are developed on the surface layer of high-speed steel under the temperature above 550°C that resulted in coagulation of special carbides lowering hardness, surface oxidation and decarbonizing.

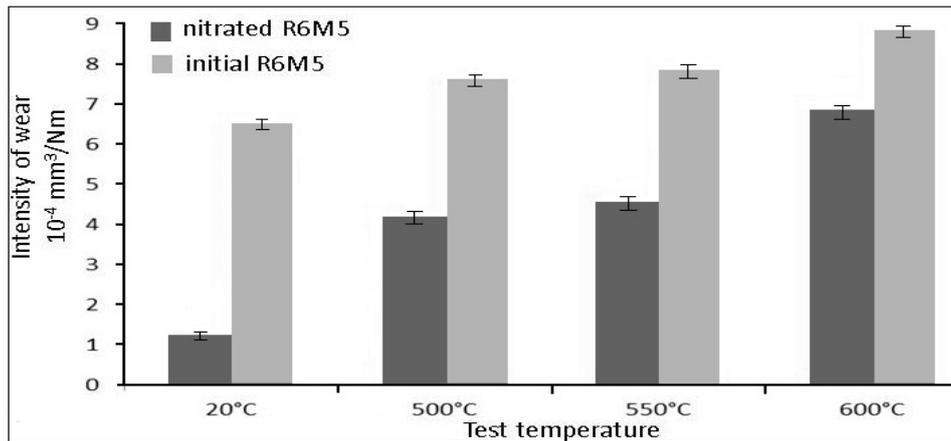


Figure 6. Dependence of steel samples' wear intensity on testing temperature

Comparison of wear intensity and friction coefficient of nitrated layers for different testing temperatures (500 °C, 550 °C and 600 °C) gives ground to conclude that wear resistance of nitrated layer of high-speed steel changes depending on ambient temperature i.e. operation temperature and nitrated layer is more stable up to $T = 550$ °C. Temperature raise up to 600 °C leads to degradation in wear resistance and increase in friction coefficient. So, it can be stated that the formation of nitrated martensite with higher thermal capacity in comparison with iron and having fine nitrides is the main factor responsible for wear resistance of nitrated layers under high temperatures [11, 12].

According to given result it can conclude as follows:

The best mode of electrolyte-plasma nitriding in carbamide-based electrolyte for high-speed R6M5 steel is determined. This mode enables to saturate the surface with nitrogen from low-temperature plasma and produce modified ultra-solid and wear-resistant layer.

It is found that the wear resistance of nitrated steel is changed depending on the ambient temperature i.e. operation temperature and nitrated layer is more resistant against the wear up to $T = 550$ °C. Temperature raise up to 600 °C leads to degradation in wear resistance and increase in friction coefficient. So, it can be stated that the formation of nitrated martensite with higher thermal capacity in comparison with iron and having fine nitrides is the main factor responsible for wear resistance of nitrated layers under high temperatures.

As a nutshell, considerable increase in wear resistance of high-speed steel after electrolyte-plasma nitriding confirms a potential of this method to improve aggressiveness of cutting tools made of high-speed steel.

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Б.К. Рахадиллов, Л.Г. Журерова, М. Шеффлер, А.К. Хасенов

Плазмалық азоттау кезіндегі жылдам кескіш болаттың жоғары температуралы тозуға төзімділігінің өзгеруі

Мақалада электролиттік плазмада азотталған жылдам кескіш болаттардың беттік қабаттарының трибологиялық зерттеу нәтижелері келтірілген. Сынақ «шар-диск» үлгісі бойынша 500 °С, 550 °С және 600 °С температураларында жүзеге асты. Тозуға төзімділік үйкеліс коэффициенті мен тозу қарқындылығы арқылы бағаланды. Жылдам кескіш болаттың азотталған қабатының тозуға төзімділігі қоршаған ортаның температурасына тәуелді өзгеретіндігі және азотталған қабаттың 550 °С температураға дейін тозуға төзімдірек болатындығы анықталды. Сынақ температурасын 600 °С дейін арттыру тозуға төзімділіктің төмендеуіне, ал үйкеліс коэффициентінің артуына әкелетіндігі анықталды. Электролиттік плазмалық азоттаудан кейінгі жылдам кескіш болаттардың беттік қабаттарының тозуға төзімділігінің артуына септігін тигізетін негізгі факторлар талқыланды. Карбамид негізіндегі электролитте R6M5 жылдам кескіш болатын электролиттік плазмалық азоттаудың тиімді тәртібі тәжірибе жүзінде анықталды. Оның тозуға төзімділігі мен беріктігі жоғары модификацияланған бетті алуға және бетті төмен температуралы плазмадан азотпен қанықтыруға әкелетіндігі анықталды. Жоғары температураларда азотталған қабаттардың тозуға төзімділігіне жауапты негізгі фактор темірмен салыстырғандағы жылу мөлшері көбірек болатын азотты мартенситтің және ұсақ дисперсті нитридтік бөлшектердің түзілуі болып табылатындығы анықталды.

Кілт сөздер: трибологиялық сынақ, тозу, үйкелу, азоттау, электролиттік плазмалық өңдеу, R6M5 жылдам кескіш болат.

Б.К. Рахадиллов, Л.Г. Журерова, М. Шеффлер, А.К. Хасенов

Изменение высокотемпературной износостойкости быстрорежущей стали при плазменном азотировании

В работе описаны результаты трибологических испытаний поверхностного слоя быстрорежущих сталей, азотированных в электролитной плазме. Испытания проводились при температурах 500 °С, 550 °С и 600 °С по схеме «шар-диск». Износостойкость оценивалась интенсивностью изнашивания и коэффициентом трения. Установлено, что износостойкость азотированного слоя быстрорежущей стали изменяется в зависимости от температуры окружающей среды и азотированный слой более устойчив к износу до температур 550 °С. Определено, что повышение температуры испытаний до 600 °С приводит к снижению износостойкости, а также увеличению коэффициента трения. Обсуждаются основные факторы, влияющие на повышение износостойкости поверхностного слоя быстрорежущей стали после электролитно-плазменного азотирования. Экспериментально установлен оптимальный режим электролитно-плазменного азотирования быстрорежущей стали R6M5 в электролите на основе карбамида, который позволяет проводить насыщение поверхности азотом из низкотемпературной плазмы и получить модифицированный слой высокой твердости и износостойкости. Установлено, что основным фактором, отвечающим за износостойкость азотированных слоев при высоких температурах, является образование азотистого мартенсита, который обладает большей теплоемкостью по сравнению с железом, и мелкодисперсных нитридных частиц.

Ключевые слова: трибологическое испытание, износ, трение, азотирование, электролитно-плазменная обработка, быстрорежущая сталь R6M5.

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