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# Fuel of the keeping needs of the energy discharge during nitrogen on the tribological characteristics of the design steel 45

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## Abstract

The effect of power discharge category W in BATR on tribological characteristics of nitrous steel 45 is investigated in the work. The highest wear resistance of steel 45 corresponds to mode 7, which provides optimum physico-chemical characteristics of the nitrided layer.

The ratio of the intensities of the passage of the main BATR subprocesses determines the structure and phase composition of the nitrided layers. Depending on the current combination of parameters of the mode of formation of the nitrided layer, the intensity of the flow of the above subprocesses (nitride formation, sputtering and diffusion saturation of the surface with nitrogen), and therefore the intensity of the formation of certain phases may be different, and sometimes reverse. For example, as the energy of the incident stream W increases, the pre-formed nitride layer is sprayed, which stimulates the process of nitrogen diffusion into the metal base and the formation of $\varepsilon$ and $\gamma$ - phases. When the flow energy is insufficient to atomize the nitride layer, it acts as a kind of barrier that impedes or completely stops the process of nitrogen diffusion.

It is established that the decrease in the specific discharge power leads to a decrease in the thickness of the nitride and diffusion zones and, as a consequence, the tribological characteristics deteriorate. It is revealed that at the maximum values of energy parameters, a nitrous containing layer is formed $\varepsilon$, $\gamma$ i $\alpha$ - phase. The decrease in voltage and current density causes the particle to increase $\gamma$ - pfase ($Fe_4N$) and according to the reduction of the share $\varepsilon$ - phase ($Fe_2N$). At minimum values of energy parameters of formation of nitrides on the surface is absent and the nitrided layer consists only of $\alpha$ - phase.

**Key words**: nitriding in glow discharge, current density, voltage, dry friction, specific power of the energy discharge in the chamber.

## Introduction

Particularly important in solving the problem of increasing the reliability and durability of machinery and equipment is the issue of friction and wear, as 85 … 90 % of machines fail due to the wear of the surfaces of individual parts and components. Machine-related friction and wear losses amount to 8 % of national revenue [1]. At the same time, according to the conducted research, 1/4 to 1/3 of all energy produced during the year in the world is spent on overcoming friction forces in moving joints of machines [2].

In order to increase the wear resistance of parts and components, the use of controlled surface modification methods based on the action of concentrated energy and substance fluxes on metallic surfaces has recently been preferred - vacuum, ionic and laser technologies. Among the methods of surface hardening of steels, one of the most developed and widely used is the method of anhydrous nitriding in glow discharge (BATR). Keeping all the major benefits of nitriding in water-intensive environments (ammonia, nitrogen and hydrogen), BATR enhances the plastic properties of the surface layer due to the absence of hydrogen embrittlement, lowers energy and material costs, improves working conditions (eliminates the possibility of explosion of hydrogen mixture) and is ecological.

It should also be noted that the influence of controlled parameters on the results of BATR was studied by many scientists, but energy parameters (specific energy flux density, voltage and current density in the gas chamber) were practically not considered.

Ignoring the basic energy parameters of the glow discharge, which acts as an identifier of elementary subprocesses (nitride formation, sputtering and diffusion saturation of the surface of nitrogen), which are responsible for the formation of the nitrided layer, its structure and phase composition essentially means the loss of unique control capabilities and control of process processes.

**The goal of the work** – to study the influence of the energy parameters of the BATR process on the phase composition, thickness of the nitride and diffusion zones, and tribological characteristics of nitrous structural steels.

## Research methods

The BATR was carried out at an installation setting designed that was manufactured at the Khmelnytsky National University research base. The UATR-1 installation is a diode type installation operating on direct current in anhydrous gas environments (nitrogen and argon). The installation is additionally completed with heating elements housed in the gas discharge chamber, which allows to change the voltage value in the chamber at a given surface temperature of the parts (samples) [3].

Based on the experience of experimental and production practice, the following BATR parameters were adopted to optimize the number of experiments: temperature $T$ = 833 K, nitriding time - 4 h, composition of gas mixture 80 % $N_2$ + 20 % $Ar$. An arbitrary voltage value was chosen, and the current density ($j = I / S$, where $I$ is the current strength, A; $S$ is the surface area of the cathode, which is equal to the sum of the suspension and sample areas, $m^2$) was determined by a combination of arbitrarily specified voltage and gas mixture pressure. Also found specific power of electric discharge in the discharge chamber $W = UI / S$, $kW / m^2$. The values of the BATR parameters are shown in table 1.

Table 1

**BATR modes with independent saturation parameters**

| Mode | 1* | 2 | 3 | 4* | 5 | 6 | 7* | 8 | 9 |
|---|---|---|---|---|---|---|---|---|---|
| Pressure $P$, Pa | 53,2 | | | 106,4 | | | 159,6 | | |
| High-voltage $U$, V | 1100 | 820 | 515 | 840 | 515 | 300 | 700 | 515 | 300 |
| Current density $j$, $A/m^2$ | 11,0 | 7,2 | 3,2 | 13,2 | 7,2 | 2,8 | 15,8 | 12,8 | 7,2 |
| Specific power $W$, $kW/m^2$ | 12,2 | 5,9 | 1,65 | 11,1 | 3,71 | 0,84 | 11,1 | 6,59 | 2,2 |

Note: *Modes carried out with dependent nitriding parameters (without additional heating of samples).

Constructional steel 45 was tested. The determination of the elemental composition of the steels was performed on an energy dispersive X-ray fluorescence spectrometer with an SSD detector X-123 (Amptek, USA). The identification of steel grades was carried out by identifying eight chemical elements (C, Al, Si, P, S, Ti, Cr, Mn). Control of the chemical composition showed that the tested samples meet the existing standards.

The surface microhardness was measured with a PMT-3 microhardness meter.

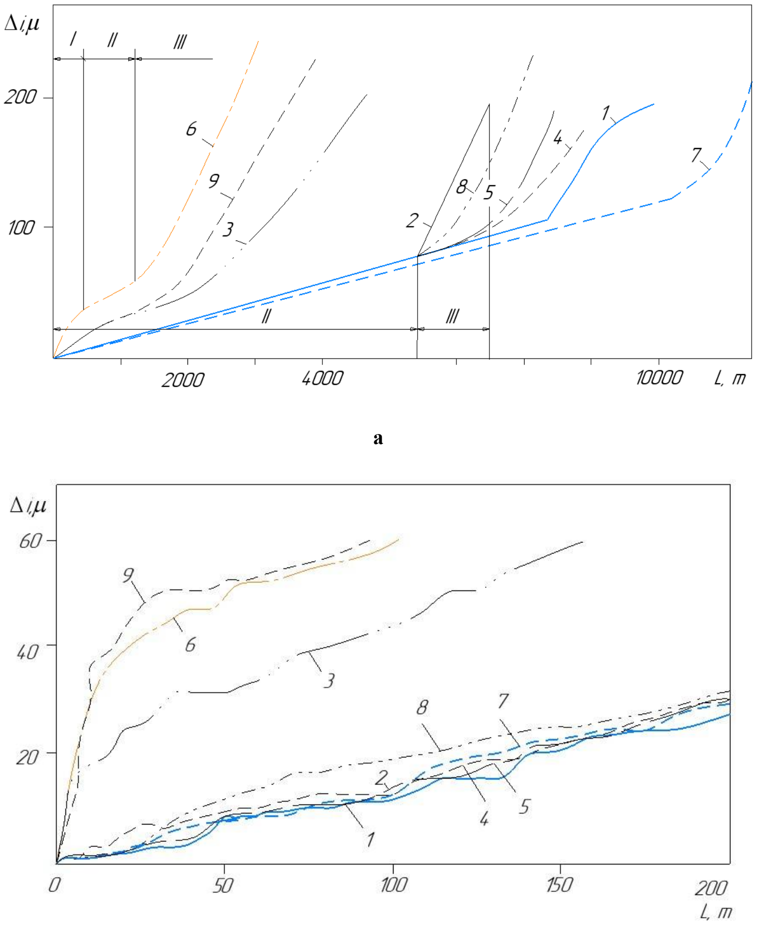
The etching of the steels to detect the structure of the nitrated layer was carried out in 3 % alcoholic nitric acid (HNO3) solution. The thickness of the nitride zone was found using a MIM-10 metallographic microscope.

The X-ray diffraction analysis was performed on a DRON-3 diffractometer in the filtered radiation of an anode in the range of angles $\theta = 20^\circ \dots 100^\circ$ with a scan step of 0.1 and an exposure time of 10 s.

The study of samples for wear resistance in dry friction was carried out on a universal machine 2168УМТ.

## Results of the experiments and the discussion

The dependence of the wear resistance of the modified BATR surfaces on the nitriding modes was confirmed as a result of tribological tests. Thus, in the conditions of dry friction, the wear intensity $I_h = \Delta i/L$ (the angle of inclination of the curves to the abscissa axis) decreases for the surfaces modified at higher values of the specific power of the energy flow $W$ (Fig. 1).



a

b

**Fig. 1. Wear (a) and ageing (b) curves of nitrous steel 45**
**(the figures on the curves correspond to the batter modes that are shown in table 1)**

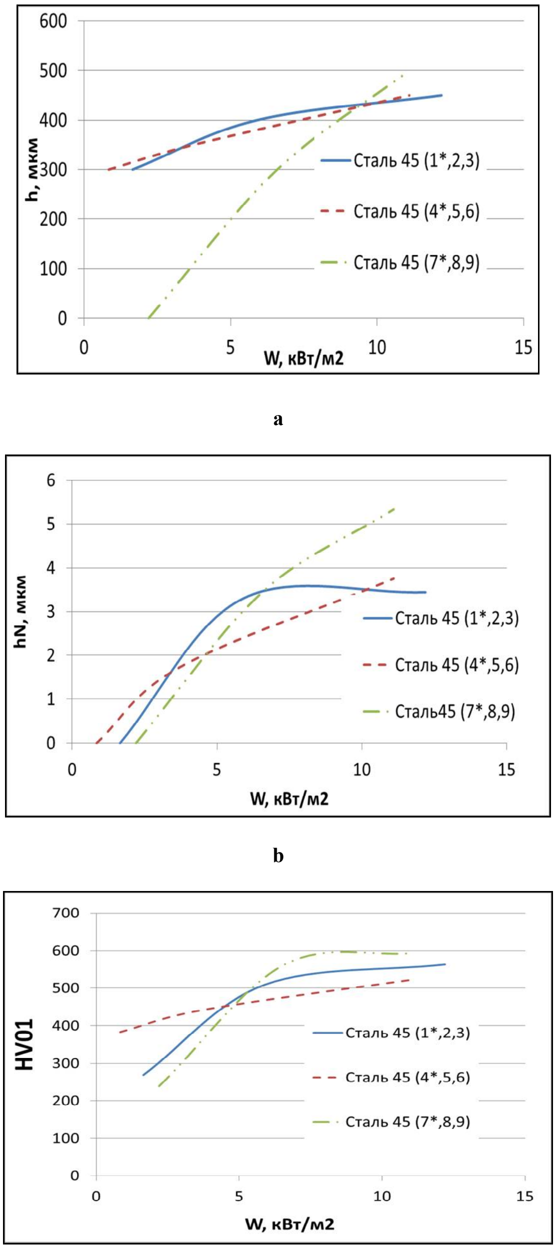
Figure 1, and shows the wear zones of nitrous steel 45 for modes 6, 3 and 9: I – aging zone; II – zones of established and III – catastrophic wear. For other modes we have two wear zones: II and III. The placement of curves 6, 9, 3 on the wear and tear graphs (Fig. 1, a and b) correspond to the lowest values of the specific energy density W = 0.84; 2.12 and 1.65 kW / m2 respectively (Table 1).

Thus, modes 6, 9, and 3 for carbon steels (steel 45) showed the worst performance in wear resistance compared to other modes studied. The maximum length of the fixed wear zone for steel 45 ranges from 650 to 1000 m (modes 1 and 7). These BATR modes are characterized by the highest values of $W$ = 12.2 kW / m$^2$ (mode 1) and $W$ = 11.1 kW / m$^2$ (modes 4 and 7). Therefore, the value of the specific power of the energy flow in the discharge chamber significantly affects the tribological characteristics of steels. Obviously, this influence is manifested by the formation of the appropriate structural and phase composition of the nitrided layers on the investigated steel, which determine its physicochemical and tribological characteristics.

In [4], the BATR energy parameters were named "the most important factors for controlling diffusion saturation under conditions of the existence of a glow discharge" and it was proposed to use the specific power of the glow discharge W. as the energy criterion.

Studies of the effect of W on the diffusion thicknesses of h and nitride hN layers and the surface microhardness of $HV_{01}$ are closely related. As W increases, the value of h, $h_N$ and $HV_{01}$ increases (Fig. 2).

The greatest thickness of the modified (diffusion) layer is obtained on carbon steel 45, (Fig. 2, a), and on alloy steels it is smaller. In this case, for modes 7, 8 and 9, we have a curve of change in the thickness h of the diffusion zone starting from zero at $W$ = 2.2 kW / m$^2$ (mode 9) to a maximum value of $h$ = 500 μm at $W$ = 11.1 kW / m$^2$ (mode 7). The maximum value of the nitride zone hN (Fig. 2, b), as well as the surface microhardness, also corresponds to mode 7 (Fig. 2, c). Accordingly, the highest wear resistance of steel 45 corresponds to the nitriding mode 7. (Fig. 1, a).



**a**

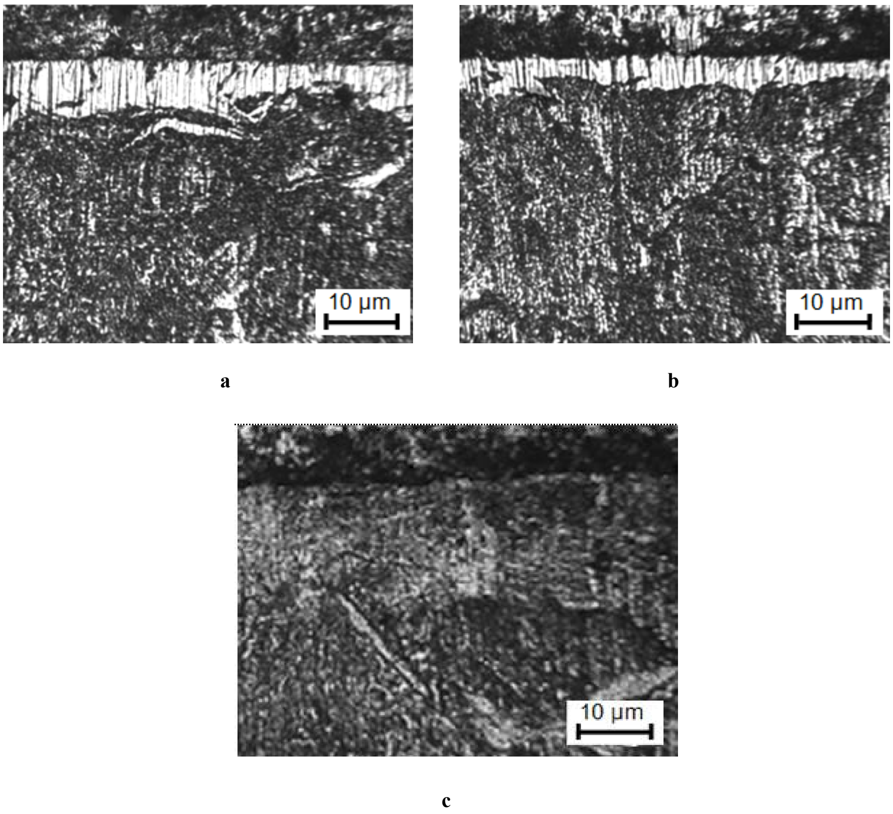
**b**

**c**

**Fig. 2. Change dependence:**
**a – thickness h of diffusion zone;**
**b – hN - nitride zone;**
**c – surface microhardness HV01 from W after BATR of steel 45**
**(curves constructed according to table 1)**

Similarly, the value of surface microhardness changes. The worst results were obtained for modes 3, 6 and 9 in which there is no nitride zone at all and the microhardness of the surface approaches the microhardness of the substrate. If for modes 3 $W$ = 1,65 kW / m$^2$ and 6 $W$ = 0,84 kW / m$^2$, the presence of a diffusion layer on steel 45 is characteristic, then for mode 9 with a higher specific power $W$ = 2,2 kW / m$^2$, the absence of a nitrogenized layer at all needs a separate explanation.

It is known [3, 5] that at BATR the main competing, complementary and mutually contradictory subprocesses pass simultaneously: nitride formation, sputtering and diffusion saturation of the surface with nitrogen. The energy conditions for the passage of the main subprocesses differ significantly: yes, nitride formation occurs at low values of the specific power of the energy flow, and the surface sputtering process is activated at high voltage values. Since mode 9 has the lowest voltage $U$ = 300 V, obviously, the sputtering process does not occur and there is no diffusion of nitrogen into the depth of the metal surface. The latter is convincingly confirmed by metallographic analysis of etched micro-grinders of steel 45 (Fig. 3).



a b

c

**Fig. 3. Micro-grinders of nitrous steel 45 (light band - nitride zone):**
**a – mode 7;**
**b – mode 8;**
**c – mode 9**

It can be seen from Fig. 3 that when the BATR is in mode 9, the nitride zone is not formed on steels, the microhardness measurements also confirm this. The microhardness on the surface was HV$_{01}$ = 238, within the distance of 200 … 1000 μm HV01 = 243 … 240.

The decryption of the diffraction pattern of a sample of 40X nitrided by mode 9 also showed the absence $\varepsilon$ and $\gamma$ - fase.

In addition, mode 9 is implemented at a maximum pressure $P$ = P3 = 159,6 PA (table 1), which caused the absence of a glow discharge in the nitriding process.

Modes 3 and 6, despite the insufficient values of $W$, pass at lower pressures of the gas mixture, and therefore a sub-process of sputtering is realized, but there is no subsequent diffusion of nitrogen atoms.

The ratio of the intensities of the passage of the main BATR subprocesses determines the structure and phase composition of the nitrided layers. Depending on the current combination of parameters of the mode of formation of the nitrided layer, the intensity of the flow of the above subprocesses, and therefore the intensity of the formation of certain phases may be different, and sometimes the reverse. For example, as the energy of the incident flux W increases, the pre-formed nitride layer is sprayed, which stimulates the process of nitrogen diffusion into the thickness of the metal base and the formation of $\gamma$ and $\alpha$ - phases. When the flow energy is insufficient to spray the nitride layer, it acts as a kind of barrier that impedes or completely stops the nitrogen diffusion process. The latter factor also explains the low rates of $h$, hN, and HV01, as well as the low tribological rates obtained in studies in modes 3, 6, and 9.

In [6], it is argued that there is an extreme relationship between the specific power of the glow discharge $W$, called the load-bearing capacity criterion of a gas medium, and its overall pressure. The authors of the paper argue that the pressure of the gas medium, which corresponds to the maximum specific discharge power, provides obtaining the nitrided layer of the greatest under the given conditions of thickness.

The results of our studies (Fig. 4) showed the absence of such dependence. On the contrary, as the pressure $P$ increases, the gas mixture W decreases (curves I and II in Fig. 4), and with independent nitriding parameters (without chamber heating), the pressure dependences $W$ have a completely different appearance. When $U = \text{const}$ (modes 3, 5 and 8, table 1) with increasing pressure, the current density j in the discharge chamber increases and, conversely, at $j = \text{const}$ (modes 2, 5 and 9, table 1) increase the pressure of the gas mixture leads to a decrease in voltage. Similar dependencies in the modes of thermostabilization were obtained in [3].

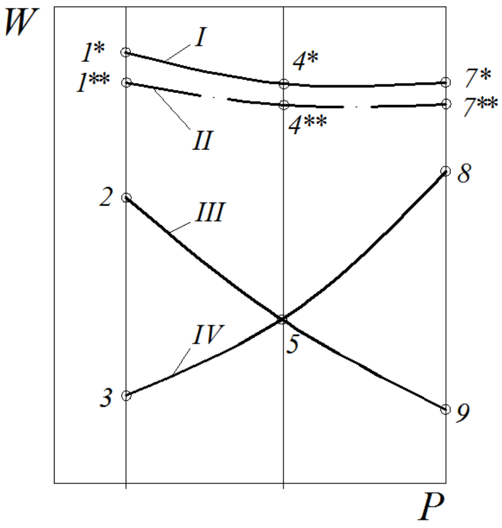


**Fig. 4. Dependence of the specific power of the discharge *W* on the pressure of the gas mixture:**
**I, II – without additional heating;**
**III, IV – offline (with optional camera heater)**

The latter confirms that even in those studies where energy parameters are considered, there are insufficient specific data to make unambiguous conclusions about their effect on the physicochemical characteristics of the nitrous layer. Thus, the results of studies of the average energy of ions with voltage and pressure do not allow them to be considered as the basis for establishing the regularities due to the absence of the composition of the gas mixture, as well as the way of changing the voltage, subject to constant values of temperature and pressure. The same applies to the results reported in [8, 9], where the effect of current density on the thickness of the nitrided layer was investigated. According to [9], increasing the current density leads to an increase in the thickness of the nitrided layer, and according to [8] – to its decrease.

The analysis of the influence of BATR parameters on the results of nitriding indicates their interdependence. Thus, the composition of the gas mixture and its pressure affect the voltage of the appearance of the glow discharge, and the change in voltage and current density, determines the saturation temperature and pressure of the gas medium [4].

Obviously, while ensuring the independence of the BATR energy parameters, additional opportunities are opened both for the intensification of the process and for controlling the processes of formation of the required physicochemical characteristics of the nitrided layers, which correspond to the operating conditions of the parts of machines and apparatus. In this case suppression or, on the contrary, intensification of certain subprocesses and formation of different structures of the modified layer is possible [10].

Thus, the study of the impact of BATR energy parameters on its results is an important scientific challenge, which opens up new possibilities in terms of optimization of nitriding regimes, which provide the results of modification of metal surfaces that best meet the requirements of parts operation.

## Conclusions

1. The BATR process can be regulated by changing the mode parameters (temperature, composition of the gas mixture, its pressure and nitriding time) and energy (specific power of the energy flow, current density and voltage at the electrodes of the gas discharge chamber). The influence of regime parameters on their structure and phase composition and physicochemical properties of nitrided layers of the study is quite comprehensive, the influence of energy parameters was investigated only at the initial level.

2. It is established that a decrease in the specific discharge power leads to a decrease in the thickness of the nitride and diffusion zones and, as a consequence, the tribological characteristics deteriorate. It is revealed that at the maximum values of the energy parameters, a nitrous layer containing $G$ and зи phases is formed It is revealed that at the maximum values of energy parameters, a nitrous containing layer is formed $\varepsilon$, $\gamma$ i $\alpha$ - pfhase. The decrease in voltage and current density causes the particle to increase $\gamma$ - phase ($Fe_4N$) and according to the reduction of the share $\varepsilon$ - pfase ($Fe_2N$). At minimum values of energy parameters of formation of nitrides on the surface is absent and the nitrided layer consists only of $\alpha$ - pfase.

3. It has been found that in conditions of dry friction for surfaces nitrided at higher energy values, the wear rate and the path of working are reduced and the path of wear is increased..

## References

1. Шевеля В.В. Трибохмимя и реологія износостойкости: монографія / В.В. Шевеля, В.П. Олександренко. – Хмельницький: ХНУ, 2006. – 278с.

2. Сафонов Б.П. Инженерная трибология: оценка износостойкости и ресурса трибоспряжения / Б.П. Сафонов, А.В. Бегова. – Новомосковск, РХТУ, 2004. – 65с.

3. Stechyshyn M.S. Influence of the Ionic Nitriding of Steels in Glow Discharge on the Structure and Properties of the Coatings / Stechyshyn, M.S.,Martynyuk, A.V.,Bilyk, Y.M.,Oleksandrenko, V.P.,Stechyshyna, N.M.// Materials Science. – 2017. - 53 (3). - pp.343 – 349.

4. Арзамасов Б.Н. Ионная химико-термическая обработка сплавов / Б.Н. Арзамасов, А.Г. Братухин, Ю.С.Елисеев, Т.А. Панайоти. – М.: Изд-во МГУ им. Н.Э. Баумана, 1999. – 400с.

5. Pastukh I.M. Subprocesses Accomponing Nitriding in a Glow Discharge / I.M. Pastukh // Technical Physics, 2014, Vol.59, no.9. – P.1320-1325.

6. Арзамасов Б.Н. Роль удельной мощности разряда при ионной химико-термической обработке сплавов / Б.Н. Арзамасов, Т.А. Панайоти // Металловедение и термическая обработка метталов. – 2000. – №6. – С.31-34.

7. Арзамасов Б.Н. Химико-термическая обработка металов в активизированных газовых средах / Б.Н. Арзамасов. М.: Машиностроение, 1979. – 224с.

8. Effect of nitriding current density on the surface properties and crystallite size of pulsed plasma-nitrided AISI 316L / J. C. Diaz-Guillen, E.E. Granda-Gutierrez, G. Vargas-Gutierrez, M. R. Diaz-Guillen // Journal of Materials Sciences and Chemical Engineering. – 2015. – No. 3. – pp. 45–51.

9. Spalvins T. Advances and Direction of Ion Nitriding / T. Spalvins // 2nd Ion Nitriding Conference, Ohio, September 18-20, 1989. Ohio, 1989. – P. 3–11.

10. Stechyshyn, M.S., Stechyshyna, N.M., Martynyuk, A.V., Luk'yanyuk, M.M.Strength and Plasticity of the Surface Layers of Metals Nitrided in Glow Discharge.(2018) Materials Science, . Article in Press.DOI: 10.1007/s11003-018-0156-5.

**Стечишина Н.М., Стечишин М.С., Мартинюк А. В.** Вплив питомої потужності енергетичного розряду при азотуванні на трибологічні характеристики конструкційної сталі 45.

У роботі досліджено вплив потужності енергетичного розряду *W* при БАТР на трибологічні характеритстики азотованої сталі 45. Встановлено, що при максимально можливій питомій потужності енергетичного розряду *W* (до його переходу в електродуговий) підвищується зносотійкість при терті. Найбільша зносотійкість сталі 45 відповідає режиму 7, який забезпечує оптимальні фізико-хімічні характеристики азотованого шару.

Співвідношенням інтенсивностей проходження основних субпроцесів БАТР визначається структура та фазовий склад азотованих шарів. Залежно від поточної комбінації параметрів режиму формування азотованого шару інтенсивність перебігу вище вказаних субпроцесів (утворення нітридів, розпорошення і дифузійне насичення поверхні азотом), а отже й інтенсивність утворення тих чи інших фаз може бути різною, а інколи і зворотньою. Наприклад, при підвищенні енергії падаючого потоку *W* попередньо утворений шар нітридів розпорошується, а це стимулює процес дифузії азоту в товщу основи металу і утворення $\gamma$ і $\alpha$–фаз. У випадку, коли енергія потоку недостатня для розпорошення нітридного шару, він виступає у ролі своєрідного бар'єру, що перешкоджає або повністю припиняє процес дифузії азоту

Встановлено, що зменшення питомої потужності розряду приводить до зменшення товщини нітридної і дифузійної зон і, як наслідок, погіршуються трибологічні характеристики. Виявлено, що при максимальних значеннях енергетичних параметрів формується азотований шар, що містить $\varepsilon$ , $\gamma$ і $\alpha$–фази. Зменшення напруги і густини струму приводить до збільшення частки $\gamma$-фази ($Fe_4N$) і відповідно до зменшення частки $\varepsilon$ – фази ($Fe_2N$). При мінімальних значеннях енергетичних параметрів утворення нітридів на поверхні відсутнє і азотований шар складається лише з $\alpha$–фази.

**Ключові слова**: азотування в тліючому розряді, густина струму, напруження, сухе тертя, питома потужність енергетичного розряду в камері.