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PHYSICO-CHEMICAL PROPERTIES AND WEAR RESISTANCE OF NITRIDED STEEL 38KhMUA

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Summary*. The influence of regime (temperature, composition of gas mixture and its pressure) and energy characteristics (voltage, current density, specific discharge power) at hydrogen-free-nitriding in a glow discharge (HFNGD) on the structure, phase composition of nitrided layers is investigated. It is shown that due to the combination of regime and energy parameters of HFNGD it is possible to achieve physical and chemical indicators of nitrided layers set by operating conditions. The set of traditionally fixed values of regime parameters (temperature, gas mixture connection, pressure and saturation time) without taking into account energy characteristics (voltage, current density and specific discharge power) significantly reduces the technological capabilities of HFNGD. With controlled regulation of the energy characteristics of HFNGD, a significant reduction in the energy consumption of the nitriding process is achieved. It is established that the energy levels of the main subprocesses differ significantly: the formation of nitrides occurs at low energies, surface sputtering is realized at high voltage values, and nitrogen diffusion occurs at high current densities. In cases where the flow energy is insufficient, either a glow discharge may not occur at all, or at insufficient stress the nitride layer on the surface is not sprayed and it acts as a barrier that prevents the diffusion process into the inner layers of the metal, leading to low physicochemical indicators of nitrided layers.*

The priority in the formation of one or another phase (ε, γ', u, α), the quantitative ratio between them and the required performance properties of the metal, respectively, can be achieved only through an independent combination of energy and regime characteristics of HFNGD.

Key words*: hydrogen-free-nitriding in a glow discharge (HFNGD), phase composition, voltage, current density, specific discharge power.*

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Statement of the problem. One of the modern and effective methods of hardening metals is nitriding in a glow discharge in an ammonia medium or in hydrogen-free medium (nitrogen + argon) – HFNGD (hydrogen-free nitriding in a glow discharge). Nitriding in hydrogen-free medium eliminates the possibility of explosion of the installation and hydrogen embrittlement of surfaces due to the diffusion of hydrogen formed during decomposition of ammonia to the depth of the metal. In addition, the HFNGD process is completely environmentally friendly.

Analysis of recent research. Nowadays, most studies are devoted to nitriding processes with interdependent parameters, i.e. each combination of mode parameters (temperature, composition and pressure of gas mixture) automatically corresponds to combination of energy (voltage, current density, specific discharge power), so the latter are not considered as controlled factors of the process [1].

In [1–3] the issues of influence of nitriding regimes (without taking into account the energy characteristics of saturation process) on the structure, phase composition, physicochemical properties of nitrided layers are widely studied.

Among all the mode characteristics of the process, the most important parameter is the set surface temperature, because to achieve and maintain it throughout the nitriding time requires a specific combination of voltage and current density. Providing a given surface

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temperature due to alternative to the glow discharge factors, allows to change the energy parameters of HFNGD in a wider range. In world practice, ensuring the independence of temperature from the energy characteristics of HFNGD is achieved by using cameras with «hot walls» [4]. Ensuring a given surface temperature due to alternative to glow discharge factors not only reduces the nitriding time, but also opens up fundamentally new opportunities to improve the controllability of the HFNGD process and obtain surface properties of metallic materials depending on their operating conditions. Despite the obvious new possibilities of HFNGD with independent energy parameters, it has not actually been studied in practical and experimental aspects.

This paper presents the results of experimental studies, comparing the results of physicochemical properties of nitrided surfaces obtained by nitriding with autonomous and interdependent HFNGD modes.

Objective of the research is to study the influence of energy parameters on the physicochemical and tribological properties of diffusion layers on the example of 438KHMUA steel at HFNGD with interdependent and autonomous saturation parameters.

Statement of the problem. The influence of HFNGD energy parameters on the depth of nitride and diffusion layers, their phase composition, microhardness and wear resistance at dry friction was studied on the example of 38HMYUA steel.

Nitriding was carried out at the UATR-1 installation designed and manufactured at the Podilsk Scientific Physical and Technological Center (PNPC) of Khmelnytsky National University. The installation belongs to the diode type model on direct current and has been additionally completed with heating elements placed in the gas-discharge chamber which enabled to change arbitrarily value of voltage U, and current density value j was determined as the relation of voltage to the total area of charge and suspension parts (samples) ($j=I/S$, A/м^2). The design features of the installation for HFNGD processes in interdependent and autonomous nitriding modes are described in detail in [5].

Nitriding modes. The influence of temperature, composition of the gaseous medium, its pressure and saturation time on the structure, phase composition and, accordingly, on the physicochemical properties of the surface nitrided layers, have been comprehensively studied, for example, in [6–7]. Therefore, nitriding was performed in a mixture of 80% N_2 (nitrogen) and 20% Ar (argon) at the temperature T = 833K for $\tau = 4$ h. The voltage and current were chosen arbitrarily, based on the experience of previous studies. Technological modes of HFNGD processes are given in Table 1.

Table 1

Characteristics of HFNGD modes

Mode	*1*/1***	*2*	*3*	*4*/4***	*5*	*6*	*7*/7***	*8*	*9*
*Pressure, **p**, Pa*	*53.2*			*106.4*			*159.6*		
*Voltage, **U**, B*	*1100/680*	*820*	*515*	*840/610*	*515*	*300*	*700/540*	*515*	*300*
*Current density, **i**, A/m^2*	*11/15.3*	*7.2*	*3.2*	*13.2/16.4*	*7.2*	*2.8*	*15.8/17.2*	*12.8*	*7.2*
Thickness of nitride zone hN, μm	*7.86/5.26*	*6.27*	*0*	*8.00/2.72*	*3.20*	*0*	*7.13/5.39*	*5.96*	*0*
Thickness of diffusion layer, h, μm	*200/250*	*150*	*50*	*200/250*	*10*	*10*	*300/300*	*150*	*0*
Microhardness of the surface $HV_{0,1}$	*1058/1084*	*1041*	*282*	*1019/1067*	*360*	*331*	*1098/1151*	*641*	*263*

*nitriding with interdependent parameters, ** – also, but with the use of suspension of a different shape and size, 2, 3, 5, 6, 8, 9 – experiments with nitriding with independent energy parameters.

Specific power of the glow discharge was determined by the formula:

$$W = UI / S = Uj, \qquad (1)$$

where S is a total area of parts (samples) and suspension (cathode area).

Experiments 1^*, 4^*, 7^* were carried out under interdependent nitriding regimes (the first series of experiments), i.e. each pressure of the mixture corresponds to the respective values of voltage and current density. The second series of experiments (1^{**}, 4^{**}, 7^{**}) was performed under the same modes but with a modified suspension shape and therefore with a different cathode surface area. Experiments 2, 3, 5, 6, 8 and 9 correspond to the autonomous HFNGD modes. In this case, modes 3, 5 and 8 are carried out when $U = 515B = const$, and modes 2, 5 and 9 when $j = 7{,}2A / м^2 = const$.

Metallographic studies were carried out on a MIM-10 microscope after etching the microgrinds with 3% alcohol solution of nitric acid. Microhardness was determined with a microhardness tester PMT-3 at a load of 0.98 N with fixation of hardness values on the surface and at a distance of 25, 50, 100, 200, 500 μm.

The DRON-3 diffractometer in the range of angles θ from 20° to 100° with a scanning step $0{,}1^\circ$ and an exposure time of 10 s was used for X-ray phase analysis.

Tribological tests. Experimental studies of samples for wear resistance were performed on a universal machine for testing materials for friction model 2168UMT. Friction scheme – «disk – finger»; type of contact – sliding plane on the plane (the end of the cylindrical sample slides on a flat metal disk; the material of the counterbody is ШХ15 steel with a hardness of the base HRC61; pressure in the contact zone p = 16 MPa; sliding speed v = 0.1 m/s.

The controlled parameter is the linear wear *h*, which was determined as the change due to the passage of a section of length *l* of the linear size of the sample, measured along the normal to the friction surface.

Results of the research. In addition to comparing the physicochemical nitrided layers of 40X steel in HFNGD with interdependent and autonomous energy modes of processes, the aim of the research was also to test the influence of the specific power of the glow discharge w, which in [8] is called plasma energy density (PED)and where it is stated that the dependence of the gas mixture on the specific power of discharge is of extreme nature: that is, the gas mixture pressure, which corresponds to the maximum w provides a nitrided layer of the greatest depth [8]. However, research [5] and the results of our experiments (Fig. 1) do not prove the existence of such an extreme dependence.

The change in the shape of the suspension led to a change in the values of w (curves I and II in Fig. 1) and in the second case we have much lower use of energy for the nitriding process. Sharp difference of curves I and II indicates the dependence of HFNGD processes on the shape and configuration of the suspension, placement of parts on it, presence of sharp edges, recesses, holes, etc. The list of factors influencing the results of HFNGD are given in [9] and include 13 items.

However, this is not the main advantage of nitriding, and the main factor is the physico-chemical properties of the reinforced layers, which provide the specified performance of the reinforced surfaces of parts. Thus, in [10… 12] there are doubts about feasibility of using specific power as a single energy criterion due to the possibility of arbitrary combination of its values with different values of voltage and current density at constant pressure of the gas mixture. The presented studies have shown that it is more expedient to estimate changes in voltage and current density. Specific power of discharge can only serve to estimate the transition from «dark» to glowing or to electric arc discharge.

The study of volt-ampere characteristics of HFNGD with interdependent (without preheating samples) parameters showed a significant difference when changing the shape and size of the suspension (Fig. 2). Thus, the change of the suspension led to a shift of the volt-ampere characteristic of the process to the left: a decrease in voltage values led to an increase in current density with a simultaneous decrease in w (Fig. 1). At the same time, the mode parameters (pressure, temperature, mixture composition and nitriding time), as mentioned above, remained unchanged. Increasing the pressure of the mixture also leads to a decrease in voltage with a simultaneous increase in current density (Fig. 2). As the increase in the absolute value of current is much smaller compared to the absolute value of the voltage (Table 1), the result is a decrease in the value of the specific power of the energy flow *w* (Fig. 1).

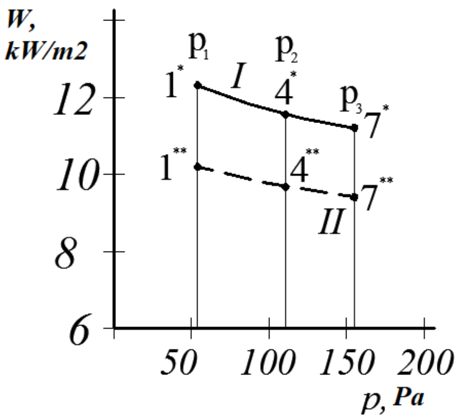

Figure 1. Dependences of specific power of the category *w* on pressure of gas mix at interdependent modes of HFNGD: I – modes 1*, 4* and 7*; II – also when changing the shape of the suspension (modes 1**, 4** and 7**)

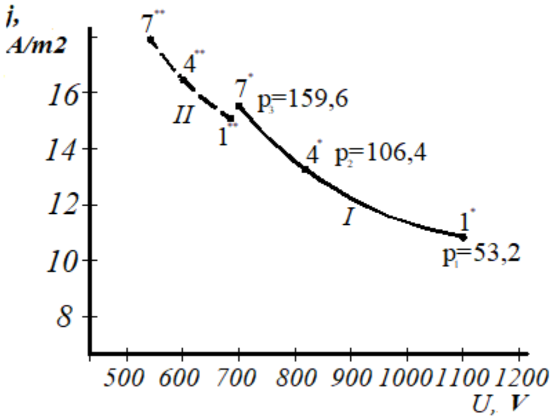

Figure 2. Volt-ampere characteristics of the HFNGD process at autonomous (interdependent) saturation modes (I and II – different shapes of suspensions)

Analysis of the experimental results shows that with decreasing energy parameters of the HFNGD process, the microhardness of the surface, the thickness of the nitride and diffusion layers decrease. X-ray diffraction analysis data also show the dependence of the structure and phase composition of the nitrided layers on the energy parameters. The energy conditions of the main HFNGD subprocesses differ significantly. Thus, the formation of nitrides occurs at low energies, while the process of spraying nitrides on the surface requires high stress values (modes with interdependent parameters), and for nitrogen diffusion into the thickness of the metal it is necessary to increase the current density. As the energy of the incident flow increases, the pre-formed layer of nitrides is sprayed, which stimulates the process of nitrogen diffusion into the depth of the metal.

When the energy flow is insufficient to spray the nitride layer, it acts as a barrier that prevents the diffusion of nitrogen. This explains the low indicators of physicochemical properties of surfaces during nitriding at modes 3, 6 and 9.

The results of tribological tests also confirm the dependence of wear resistance on the energy characteristics of HFNGD (Fig. 3).

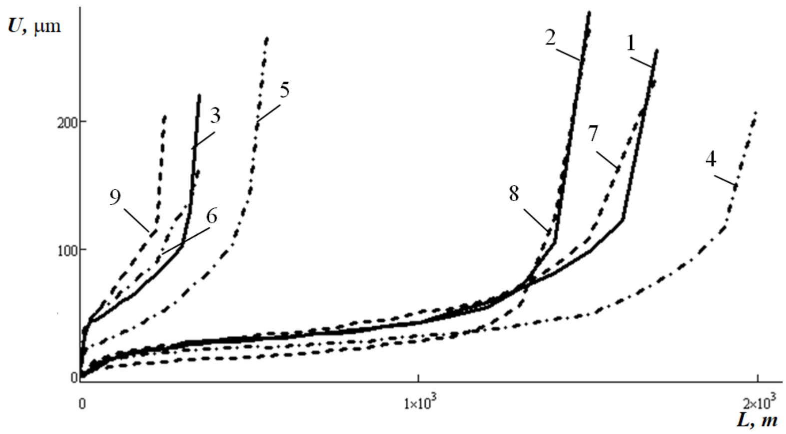

Figure 3. Wear resistance of nitrided steel 38HMYUA after HFNGD (figures on the curves correspond to the nitriding modes in Table 1)

As a result of the analysis of wear resistance data of nitrided steel 38HMYUA in the conditions of dry friction it is revealed that HFNGD at higher energy parameters provides higher indicators of wear resistance. At the same time, both the wear rate and the running-in period decrease, and the period of constant wear increases. The latter refutes K. Keller's statement about the insignificant influence of energy characteristics of discharge on the results of nitriding [10].

The above research results clearly indicate a significant effect of voltage and current density on the physicochemical and tribological properties of nitrided metal layers. Moreover, in the area of energy characteristics of the regime there is a limit below which conducting of HFNGD has no sense despite the fact that the values of the mode parameters remain unchanged (modes 3, 6 and 9).

Thus, the change in the energy characteristics of HFNGD allows to significantly expand the technological capabilities of nitriding by obtaining modified layers of a given structure and phase composition: based on nitrogenous α-solid solution with or without nitride zone, or only nitride zone with ε- or only with γ′- phases. Hence, the nitride zone containing ε-phase has high corrosion resistance, and the area consisting of the γ′-phase is characterized by high ductility. Single-phase nitride zone improves the mechanical properties of nitrided steels in contrast to the two-phase (ε + γ′) zone, which has increased surface brittleness. However, at high friction velocities ε + γ′ prevent metal adhesion of friction pairs. In general, the thinner the nitride zone, the more plastic is the nitrided layer, but the lower the resistance to abrasive wear, especially in dry friction.

According to the results of X-ray diffraction analysis at maximum values of energy parameters (all modes with interdependent parameters $1^*/1^{**}$, $4^*/4^{**}$, $7^*/7^{**}$, mode 8 and partly 5 with autonomous saturation parameters) a nitrided layer is formed, which consists of ε (Fe_2N), γ' (Fe_4N) and α (Me [N]) – phases (mode 7). The decrease in voltage and current density leads to an increase in the $\gamma' -$ phase fraction in the nitride zone of the nitrided layer and, accordingly, to a decrease in $\varepsilon -$phase (modes 7, 8). At the minimum values of energy characteristics, the nitride layer is not formed on the surface and it consists only of $\alpha -$phase (mode 9, Table 1). Corrosive [11], fatigue characteristics [12], residual compressive stresses [13], strength and ductility characteristics [14] of nitrided layers change respectively.

Conclusions. The set of traditionally fixed values of regime parameters (temperature, gas mixture, pressure and saturation time) without taking into account energy characteristics (voltage, current density and specific discharge power) significantly reduces the technological capabilities of HFNGD. With controlled regulation of energy characteristics of HFNGD, a significant reduction in the energy consumption of the nitriding process is achieved.

The energy levels of the main subprocesses differ significantly: nitrides formation occurs at low energies, surface spraying is realized at high voltage values, and nitrogen diffusion occurs at high current densities. In cases where the flow energy is insufficient, either a glow discharge may not occur at all, or at insufficient stress the nitride layer on the surface is not sprayed and it acts as a barrier that prevents diffusion into the inner layers of the metal, leading to low physicochemical indicators of nitrided layers.

Priority in the formation of a phase (ε, γ', u, α), the quantitative relationship between them and the required performance properties of the metal, respectively, can be achieved only through an independent combination of energy and regime characteristics of HFNGD.

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ФІЗИКО-ХІМІЧНІ ВЛАСТИВОСТІ ТА ЗНОСОСТІЙКІСТЬ АЗОТОВАНОЇ СТАЛІ 38KHMUA

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Резюме. *Досліджено вплив режимних (температура, склад газової суміші її тиску) та енергетичних характеристик (напруга, щільність струму, питома потужність розряду) при безводневому азотуванні в тліючому розряді (HFNGD) на структуру, фазовий склад азотованих шарів. Показано, що за рахунок комбінації режимних та енергетичних параметрів HFNGD можна досягти фізико-хімічних показників азотованих шарів, заданих умовами експлуатації. Комплекс традиційно фіксованих значень режимних параметрів (температура, сполука газової суміші, тиск і час насичення) без урахування енергетичних характеристик (напруга, щільність струму й питома потужність розряду) значно зменшує технологічні можливості HFNGD. При контрольованому регулюванні енергетичних характеристик HFNGD досягається значне зниження енергоємності процесу азотування. Встановлено, що енергетичні рівні протікання основних субпроцесів суттєво відрізняються: утворення нітридів відбувається при низьких енергіях, розпилення поверхні реалізується при високих значеннях напруги, а дифузія азоту відбувається при підвищених величинах щільності струму. У випадках, коли енергія потоку недостатня може або взагалі не виникати тліючий розряд, або при недостатній напрузі шар нітридів на поверхні не розпорошується і він виступає в ролі бар'єра, який перешкоджає процесу дифузії у внутрішні шари металу, що призводить до низьких фізико-хімічних показників азотованих шарів. Пріоритет у формуванні тієї або іншої фази (*ε*,* γ'*,* u*,* α*), кількісного співвідношення між ними й необхідними експлуатаційними властивостями металу відповідно, можна досягти тільки за рахунок незалежного комбінування енергетичних і режимних характеристик HFNGD. Зниження напруги й щільності струму призводить до збільшення частки* $\gamma' -$ *фази в нітридній зоні азотованого шару й відповідно до зменшення кількості* $\varepsilon -$ *фази*)*. При мінімальних значеннях енергетичних характеристик нітридний шар на поверхні не утворюється й він складається тільки з* $\alpha -$ *фази.*

Ключові слова: *безводневе азотування в тліючому розряді (HFNGD), фазовий склад, напруга, щільність струму, питома потужність розряду.*

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