



Enhancement of corrosion–mechanical wear resistance of metallic alloys by glow-discharge nitriding

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Abstract

The aim of this work is to provide a comprehensive analysis of hydrogen-free nitriding and nitrocarburizing processes in a glow discharge, to summarize the regularities of nitride and carbonitride layer formation on steels and titanium alloys, and to evaluate their corrosion–mechanical wear resistance under conditions of sliding, fretting, cavitation, and exposure to aggressive media. Particular attention is given to the application of Pastukh's I.M. model for interpreting the kinetics and energy mechanisms of diffusion processes. The study presents a comparative analysis of the structural–phase transformations during nitriding and nitrocarburizing in a hydrogen-free glow discharge and systematizes experimental data on microhardness, electrochemical behavior, and corrosion–mechanical wear resistance.

The methodological basis of the theoretical part is the energy-exchange model developed by I. M. Pastukh, which describes the influence of active plasma species on the diffusion rate and phase formation. It is shown that hydrogen-free nitriding provides intensified nitrogen diffusion, the formation of stable nitrides TiN, Ti₂N, ϵ -(Fe₂-₃N), γ' -(Fe₄N), and the elimination of hydrogen embrittlement. It is established that nitrocarburizing produces multilayer carbonitride structures with hardness values of 900–1300 HV and high adhesion to the substrate. The modified layers demonstrate a 3–6-fold increase in wear resistance, a 2.5–4-fold reduction in cavitation damage, and a significant decrease in corrosion current in NaCl and NaOH solutions. A transition of the dominant failure mechanisms from brittle and adhesive to plastic-deformation dominated modes has been identified. The obtained results confirm the experimentally observed regularities of carbonitride-layer formation and demonstrate the key role of ion bombardment in generating the defect-subzone that governs diffusion kinetics and phase stability.

The findings may be applied in the design of wear-resistant machine components, medical implants, and elements of cavitation units operating in alkaline and chloride environments. Hydrogen-free nitriding and nitrocarburizing technologies are identified as highly efficient, economically feasible, and suitable for industrial implementation.

Keywords: glow discharge, hydrogen-free nitriding, nitrocarburizing, nitride layers, titanium, steel, corrosion–mechanical wear

Introduction

Surface strengthening of titanium alloys and structural materials remains one of the key directions for improving the operational reliability of machine components, technological equipment, and critical-purpose products. Among the known surface-modification methods, a special place is occupied by nitriding and nitrocarburizing, which enable the formation of hard diffusion layers with high wear resistance, corrosion resistance, and fretting resistance. The use of glow discharge significantly expands the capabilities of these processes by intensifying diffusion, activating plasma-chemical reactions, and reducing the treatment temperature.

Traditional plasma nitriding technologies are based on hydrogen-containing gas mixtures, which facilitate surface cleaning but may simultaneously induce several undesirable effects, including hydrogen uptake, embrittlement, phase-structure changes, and reduced service life of components. For this reason, considerable



attention is devoted to the development of hydrogen-free nitriding and nitrocarburizing processes, which eliminate unwanted side effects and ensure the formation of stable nitride layers with predictable properties.

In recent years, substantial experimental data have been accumulated regarding the influence of glow-discharge parameters on the structure and properties of nitride layers formed on titanium alloys and steels, as well as on the dependence of corrosion–mechanical wear resistance, fretting resistance, and wear behavior in corrosive environments on these parameters.

Glow-discharge nitrocarburizing is a surface-modification process for steels and alloys that combines the advantages of nitriding and carburizing. Its effectiveness is determined by the active interaction of nitrogen and carbon, which in the glow-discharge plasma form complex carbonitride phases characterized by high hardness, corrosion resistance, and fretting resistance.

Despite significant progress in studying nitriding and nitrocarburizing processes, a number of fundamental and applied aspects of these technologies in hydrogen-free glow discharges remain insufficiently clarified. In particular, the influence of the absence of hydrogen on the activation mechanisms of nitrogen and carbon, the formation of the defect sublayer, and the kinetics of diffusion in the near-surface region of metals require further refinement, as do the regularities of phase formation in hydrogen-free environments, including the stability of nitride and carbonitride phases and their correlation with plasma parameters.

Special attention should be devoted to correlating experimental results with the energy-exchange model developed by I. M. Pastukh, which describes the interaction of active plasma species with the metal surface, with practical validation for hydrogen-free nitrogen–carbon (N–C) media, and to understanding how the structural–phase characteristics of modified layers influence their wear resistance.

Literature review

Nitriding and nitrocarburizing in a glow discharge remain among the most effective surface-modification methods for steels and titanium alloys due to their ability to form hard, wear-resistant, and corrosion-resistant nitride and carbonitride phases. Recent review studies [1–7] indicate a significant increase in interest in hydrogen-free technologies, which eliminate the problem of hydrogen embrittlement and provide more intensive ionic activation of the surface.

In study [1], it was shown that low-temperature nitriding of titanium in a glow discharge leads to the formation of TiN–Ti₂N layers with a pronounced microhardness gradient, where ion bombardment plays a key role by determining defect formation and the initial stage of diffusion. Works [2–5] investigated the influence of the main glow-discharge nitriding parameters—pressure, gas composition, temperature, and treatment duration—on the evolution of surface structure and properties of stainless steels. The authors demonstrate that variations in nitriding regimes control the nitrogen diffusion rate, the formation of nitride and nitrogen-enriched solid-solution layers, as well as the final performance characteristics of the material, including hardness, wear resistance, and corrosion resistance. Accurate control of process parameters is essential for obtaining stable and optimized properties of nitrided steels, which aligns with current trends in enhancing the efficiency of glow-discharge surface modification. The research demonstrates that discharge parameters significantly influence the phase composition of layers on stainless steels. A correlation between discharge power and the ϵ/γ' -phase ratio was identified, which is consistent with the energy-exchange model proposed by I. M. Pastukh.

Study [6] emphasizes that not only the external processing parameters (pressure, gas composition, voltage, time) determine the final surface properties, but also the quantitative characteristics of the flux and dose of plasma particles that actually reach the treated surface. Article [7] shows that for titanium alloys, glow discharge provides deep nitrogen saturation without overheating the material, which is particularly important for medical implants.

A comprehensive analysis of the influence of hydrogen-free nitrogen–carbon media on titanium alloys was performed in study [8]. The authors demonstrated that a hydrogen-free atmosphere promotes stabilization of the Ti₂N phase and prevents degradation of α -titanium, which is typical for hydrogen-containing discharges. Several works, including [8, 9], systematized the kinetic features of nitrogen diffusion in titanium alloys using the energy model of I. M. Pastukh, which describes the interaction of the flux of active particles with the surface through the balance of ion-impact energy and defect generation. The model has received experimental confirmation in subsequent studies [10–13], highlighting its universality for hydrogen-free media.

Studies [14, 15] demonstrated that combined nitrogen and carbon saturation provides a more ductile and crack-resistant layer compared with pure nitriding. Carbon promotes the formation of a stable ϵ -phase with a modified lattice structure, reducing residual stresses and improving resistance to fretting and cavitation damage. These findings agree with modern results reported in [16], where it was shown that Fe–(N,C) carbonitrides exhibit superior tribocorrosion performance compared to nitrides.

Investigations of the corrosion–mechanical stability of plasma-modified layers in works [17, 18, 19] revealed that nitrocarburized layers on steels show significantly increased wear resistance and 1.5–3-fold higher cavitation resistance compared to conventional nitriding.

Analysis of current data confirms that hydrogen-free nitriding and nitrocarburizing in a glow discharge are promising surface-engineering methods that ensure intensive surface activation, formation of stable nitride and carbonitride phases, improved tribocorrosion and cavitation resistance, and complete elimination of hydrogen embrittlement.

Objectives of the Study

The primary objective of this study is to provide a comprehensive analysis of hydrogen-free nitriding and nitrocarburizing processes under glow-discharge conditions, with particular attention to the mechanisms of nitrogen and carbon activation, diffusion kinetics, and the formation of nitride and carbonitride layers on steels and titanium alloys. The research aims to clarify how the absence of hydrogen influences surface activation, phase stability, and defect formation, as well as to determine how these factors affect the microstructure and operational performance of the modified layers.

The study seeks to formulate well-grounded conclusions regarding the technological advantages, limitations, and practical applicability of hydrogen-free nitriding and nitrocarburizing for enhancing the durability of machine components, titanium elements, and structural units operating in aggressive chemical or dynamic environments.

Purpose of work

To determine the technological advantages and limitations of hydrogen-free nitriding and carbonitriding compared to traditional hydrogen-containing methods.

Methods

This research is based on the analysis of scientific studies published in recent years, as well as on experimental results in the fields of glow-discharge nitriding, nitrocarburizing, and surface modification of titanium and steels. The article summarizes data on the modification of the following materials: α - β titanium alloys VT3-1 and VT8, austenitic stainless steels 08Kh18N10 and 12Kh18N9T, structural steels 38KhMYuA, 40Kh, 20Kh, and carbon steels. The main variable processing parameters include: gas pressure in the vacuum chamber of 80–400 Pa; treatment temperature of 500–700 °C; gas-mixture composition (N₂, N₂ + Ar, N₂ + C); discharge voltage of 400–1200 V; current density of 1–10 mA/cm²; and treatment duration of 1–10 hours. To analyze the properties of the modified surface layer, the following methods are employed: surface microhardness measurement using a PMT-3 microhardness tester; nitride-layer thickness determination using microstructural analysis on an MIM-10 microscope; phase-composition analysis of the surface layer using a DRON-3M X-ray diffractometer; X-ray photoelectron spectroscopy (XPS); and electron Auger spectroscopy performed using a JAMP-10S scanning Auger microprobe and electrochemical examinations in chloride and alkaline environments.

Research

Activation of nitrogen in a hydrogen-free glow-discharge environment differs fundamentally from classical hydrogen-containing nitriding technologies. The absence of hydrogen alters the energy exchange at the gas–solid interface, affects the composition of active plasma species, and determines the diffusion–kinetic regularities of nitrogen penetration into the metal. Based on analytical developments by Pastukh I.M. and their further extension in a series of subsequent works [10,11], a model was substantiated that describes the mechanism of nitride layer formation in hydrogen-free gas-discharge media through a system of interrelated processes such as electron-impact activation, surface energy exchange, ion-stimulated diffusion, and the thermodynamics of nitride formation.

The absence of hydrogen leads to a sharp increase in the fraction of heavy ions N₂⁺ and N⁺, which carry high impulse energy. This enhances the bombardment of the metal surface and results in intensified surface cleaning, oxide-film destruction, formation of active adsorption centers, and active nitrogen diffusion. Studies on titanium alloys [13] and steels confirm the presence of an intensive primary stage of surface activation, during which a sharp increase in the nitrogen uptake rate is observed in the first 10–20 minutes of treatment. This effect was first quantitatively described in the works of Pastukh I.M. [10], where the dependence of the flux of active species on electron energy and cathode potential was demonstrated.

According to Pastukh's energy model, a distinctly non-uniform energy distribution of active particles is formed in the glow discharge, with a pronounced increase in their energy within the near-surface cathode sheath. Calculations showed that it is the cathode potential drop that ensures the acceleration of nitrogen ions, while the electron component of the plasma determines the intensity of molecular N₂ dissociation. Thus, the flux of active particles reaching the surface consists of N₂⁺/N⁺ ions, metastable N atoms, as well as fast neutral atoms formed due to charge exchange in the cathode sheath. This combined energetic action determines the nature of the primary activation of the metallic surface and the subsequent rate of diffusion saturation.

Under hydrogen-free glow-discharge nitriding, the process of hydrogen saturation is absent, which is confirmed by studies [8,15]. In this case, a more homogeneous composition of surface layers is formed; no black (brittle) zones typical of hydrogen-containing mixtures appear; adhesion of nitride phases to the substrate increases; and corrosion–mechanical resistance in chloride and alkaline environments improves. This is consistent with Pastukh's model [10]: under hydrogen-free conditions, the surface energy of the system increases, promoting the formation of thermodynamically stable mononitrides rather than metastable hydrogen-defective structures.

X-ray diffraction analysis of the VT8 titanium alloy showed that, depending on the nitriding regime, diffraction peaks of α -Ti, TiN (δ -phase), and Ti_2N (ε -phase) appear on the surface of the nitrided titanium alloy, whereas on the surface of the non-nitrided titanium alloy only α -Ti and β -Ti peaks are present (Fig. 1).

In a hydrogen-free glow discharge, the diffusion coefficient of nitrogen may increase by a factor of 2–4 compared to hydrogen-containing environments. This is associated with the higher energetic potential of N^+ ions, a greater number of direct impact processes, and an increased rate of vacancy generation in the near-surface zone of the metal. Studies on steels and titanium alloys (8; 22) recorded a sharp increase in microhardness and in the thickness of nitride layers specifically under hydrogen-free conditions, which fully corresponds to the predictions of Pastukh's model regarding the role of ion bombardment.

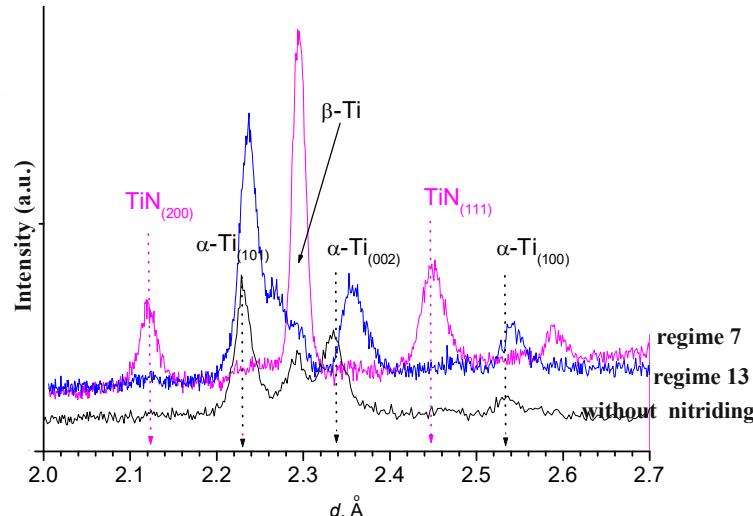


Fig.1. Radiographs for the VT8 titanium alloy: without nitriding, under the №7 regime (epi surface temperature of 660°C and pressure of 160 Pa, nitriding duration of 75 minutes) and the № 13 regime (the surface temperature of 620°C, pressure of 240 Pa, the duration of the nitriding for 20 minutes).

Based on the conducted research [15], the following regularities of nitride phase formation in hydrogen-free discharge can be distinguished: in steels, ε -(Fe_{2-3}N) and γ' -(Fe_4N) phases with high hardness of 900–1200 HV are formed; in titanium alloys, a gradient layer $\text{TiN} \rightarrow \text{Ti}_2\text{N} \rightarrow \alpha\text{-Ti}(\text{N})$ is formed [9]. Particularly important is the fact that under hydrogen-free conditions the Ti_2N phase becomes stabilized, which is an indicator of an optimal energetic regime, as experimentally confirmed in works [8,13].

After glow-discharge nitriding, the nitride layers on the surface of structural steels consist of an outer ε -phase (Fe_{2-3}N) — hard and wear-resistant; an inner γ' -phase (Fe_4N) — more ductile; and a diffusion zone with a nitrogen-graphite-type distribution. On the surface of titanium alloys, TiN (golden color), Ti_2N (light yellow), and a solid-solution zone of N in α -Ti are formed. These layers are characterized by high adhesion and thermal stability. At the same time, glow-discharge carbonitriding allows the formation of ε -($\text{Fe}_{2-3}\text{N}_{1-x}\text{C}_x$), γ' -($\text{Fe}_4\text{N}_{1-x}\text{C}_x$) phases, and complex carbonitride structures on steels. Such layers exhibit higher hardness and improved performance in highly aggressive environments.

Carbonitriding occurs in mixtures of the $\text{N}_2\text{-C}_x\text{H}_y$ or $\text{N}_2\text{-CO-CO}_2$ type, where sources of carbon are fragmented molecules of CH_4 , C_2H_2 , CO, and CO_2 in metastable states. The kinetics of carbonitriding involves simultaneous saturation of the surface with nitrogen and carbon, followed by the formation of carbonitrides $\text{Fe}(\text{C},\text{N})$ as well as a carbon-enriched ε -phase. Studies [15,22] show that under glow-discharge conditions it is possible to form a three-phase gradient layer in which the outer zone contains a carbon–nitrogen enriched layer with increased hardness up to 900–1300 HV.

The formation process of carbonitrides in a glow discharge consists of the following stages: primary cleaning and activation of the surface, bombardment by N^+ , C^+ , and N_2^+ ions, which leads to the destruction of the oxide film, the appearance of voids and vacancies, and the activation of chemisorption of C and N. The publication [22] demonstrated that ion bombardment at the initial stages creates defect subsurface zones that sharply increase the diffusion rate of carbon and nitrogen into steel. Unlike conventional gas carbonitriding, in a glow discharge this process occurs predominantly due to the ion flux rather than molecular diffusion. According to the model by Pastukh I.M., the total flux of active particles in hydrogen-free environments provides conditions under which the chemical activity of the elements exceeds the activation energy of nitride and carbonitride formation. The ε -phase $\text{Fe}_{2-3}(\text{N},\text{C})$ forms first under high surface saturation, followed by the γ' -phase $\text{Fe}_4(\text{N},\text{C})$, carbonitride inclusions in α -Fe, and nanocrystalline mixed-composition zones. It has been established that the presence of carbon stabilizes the ε -phase, increases its thickness, and prevents local failures under dynamic loading.

According to research data [15], the structure of the strengthened layer includes an outer saturated ϵ -zone, where C and N occupy mixed lattice positions; an intermediate γ' -layer with increased hardness and a quasi-monolithic structure; and a diffusion subzone with a gradual decrease in C and N concentrations. This combination ensures the absence of sharp hardness transitions, high adhesion of the layer to the substrate, low tendency to delamination, and enhanced resistance to corrosion-mechanical wear.

It has been established that hydrogen-free carbonitriding significantly reduces the coefficient of friction (down to 0.11) at a sliding speed of 0.5 m/s and a contact pressure of 12 MPa. Increasing pressure leads to plastic flow and an increase in the actual contact area on the one hand, and to deformation strengthening of the metal on the other. Although the rate of electrochemical corrosion increases in the presence of plastic deformation of surface layers, it still lags behind the rate of mechanical damage — the higher the contact pressure, the greater this difference becomes (Table 1) [17].

Table 1
Dependence of the corrosion component of damage during corrosion-mechanical wear of 40X steel in an alkaline environment ($v = 0.05$ m/s)

Pressure, MPa	Corrosion current, A/m ²	Corrosion rate, g/(m ² ·год)	Total mass loss, g/(m ² ·год)	Corrosion loss rate, %
1	0.183	0.190	0.223	85
2	0.296	0.308	0.376	82
4	0.456	0.474	0.640	74
8	2.493/0.550	2.593/0.573	5.403/0.924	48/62
12	2.718/0.970	3.027/1.010	10.095/2.062	30/49

Note: Numerator – improvement, denominator – nitriding (793 K, 75% N₂ + 25% Ar, 265 Pa + 10% propane).

Studies [15, 17] have shown that carbonitrided layers exhibit significantly increased wear resistance and resistance to corrosion-mechanical degradation. It has been established that under abrasive-corrosive and cavitation wear conditions, carbonitride layers demonstrate stability unattainable for purely nitride surfaces. This is due to the fact that the presence of carbon reduces internal stresses in the ϵ -phase, improves the plasticity of the layer, and promotes the formation of Fe(C,N) phases that are more resistant to micro-fracture [23–25].

Conclusions

1. The data obtained and summarized in this work indicate that the efficiency of hydrogen-free nitriding and carbonitriding in a glow discharge is determined by a complex interaction of plasma, surface, and diffusion processes. The modified layers produced by glow-discharge nitriding and carbonitriding exhibit high corrosion-mechanical wear resistance in all investigated environments. Glow-discharge carbonitriding ensures the formation of a multiphase strengthened layer with superior wear resistance and corrosion-mechanical durability. The combined incorporation of nitrogen and carbon stabilizes the ϵ -phase, improves the plastic characteristics of the layer, and enhances its long-term wear resistance in corrosion-active environments.

2. The experimental regularities are fully consistent with the energy model proposed by Pastukh I.M., which explains the kinetics of metal saturation with nitrogen and carbon through the balance of active particle fluxes and the energetic stability of the resulting phases, thus confirming the adequacy of the theoretical description of the processes.

3. Glow-discharge carbonitriding is an optimal strengthening method for structural steels operating under conditions of cavitation, fretting, abrasive wear, and corrosion-induced degradation.

4. Glow discharge is one of the most effective methods for producing wear-resistant, corrosion-resistant, and stable surface layers on steels and titanium alloys.

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М.С. Стечишин, Н.С. Машовець, С.Я. Підгайчук, В.М. Шевчук, А.В. Корінний Підвищення корозійно-механічної зносостійкості металевих сплавів азотуванням в тліючому розряді

Метою роботи є комплексний аналіз процесів безводневого азотування та карбоазотування у тліючому розряді, узагальнення закономірностей формування нітридних і карбонітридних шарів на сталях і титанових сплавах, а також оцінювання їх корозійно-механічної зносостійкості в умовах тертя, фретингу, кавітації та дії агресивних середовищ. Окрема увага приділена застосуванню моделі Пастуха І.М. для інтерпретації кінетики та енергетичних механізмів дифузійних процесів. У статті проведено порівняльний аналіз структурно-фазових перетворень при азотуванні і карбоазотуванні в безводневому тліючому розряді, виконано систематизацію експериментальних даних щодо мікротвердості, електрохімічних характеристик і корозійно-механічної зносостійкості. Методологічною основою теоретичної частини є енергообмінна модель Пастуха І.М., що описує вплив активних частинок плазми на швидкість дифузії та фазоутворення. Показано, що безводневе азотування забезпечує інтенсифіковану дифузію азоту, формування стабільних нітридів TiN , Ti_2N , $\epsilon-(Fe_{2-3}N)$, $\gamma'-(Fe_4N)$ та усунення водневої крихкості. Установлено, що карбоазотування створює багатошарові карбонітридні структури з твердістю 900–1300 HV та високою адгезією до основи. Модифіковані шари демонструють 3–6-кратне підвищення зносостійкості, 2,5–4-кратне зменшення кавітаційного руйнування та значне зниження корозійного струму у $NaCl$ та $NaOH$. Виявлено зміну механізмів руйнування з крихкого та адгезійного на пластично-деформаційний. Показано відповідність експериментальних закономірностей для формування карбоазотованих шарів. Доведено ключову роль іонного бомбардування у формуванні дефектної підзони, що визначає кінетику дифузії та стабільність фаз. Результати можуть бути використані при проєктуванні зносостійких елементів машин, медичних імплантів, деталей для кавітаційних вузлів, що працюють у лужних та хлоридних середовищах. Технології безводневого азотування та карбоазотування визначені як високоефективні, економічно доцільні та придатні до промислового застосування.

Ключові слова: тліючий розряд, безводневе азотування, карбоазотування, нітридні шари, титан, сталь, корозійно-механічне зношування