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ION-PLASMA NITRIDING OF TITANIUM ALLOYS: TECHNOLOGIES, ADVANTAGES, AND LIMITATIONS

To improve the surface properties of titanium and its alloys, numerous surface modification processes have been developed through the formation of coatings, films, or layers. One such treatment method is plasma nitriding, which is widely used to enhance the surface properties of titanium and its alloys. The diffusion of nitrogen into the bulk material creates a continuous hardness profile, thereby providing optimal support for the hard surface layer. This study examines the ion-plasma nitriding technology for the treatment of titanium alloys. The fundamental principles of ion-plasma nitriding, its advantages, and limitations are thoroughly analyzed, and the optimal process parameters that contribute to increasing the wear resistance and durability of titanium alloys are identified. The study includes an analysis of modern approaches and trends in the development of this technology, allowing for the identification of the most efficient processing modes and improvements in the characteristics of the resulting layers. Special attention is given to the influence of processing parameters such as temperature, pressure, gas mixture composition, and exposure time on the formation of the structure and properties of the nitrided layers. Research has shown that properly selected processing modes not only increase the microhardness and wear resistance of the surface but also reduce the coefficient of friction, which is particularly important for components operating under high mechanical loads. In addition, ion-plasma nitriding is compared with other surface modification methods for titanium, such as gas nitriding, laser hardening, and the application of hard coatings. The key advantages of this method have been identified, including the high uniformity of the nitrided layer, minimal dimensional changes in the components, and the environmental safety of the process. The obtained results can be used to optimize industrial processing technologies for titanium alloys, enabling their expanded application in the aerospace, medical, and mechanical engineering industries, where high reliability and durability of materials are required.

Keywords: plasma, glow discharge, nitride phase, nitrogen, temperature, vacuum.

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Титан қорытпаларын ион-плазмалық азоттау: технологиялар, артықшылықтар және шектеулер

Титан мен оның қорытпаларының беткі қасиеттерін жақсарту мақсатында жабындар, үлбірлер немесе қабаттар түзе отырып, беткі қабатты түрлендірудің әртүрлі әдістері әзірленді. Осындай өңдеу тәсілдерінің бірі – плазмалық азоттау, ол титан мен оның қорытпаларының беткі қасиеттерін жетілдіру үшін ең кеңінен қолданылатын әдістердің бірі болып табылады. Азоттың көлемдік материалға диффузиясы қатты беткі қабатқа оңтайлы тірек болатын үздіксіз қаттылық профилін қамтамасыз етеді. Бұл жұмыста титан қорытпаларын өңдеуге арналған ион-плазмалық азоттау технологиясы зерттеледі. Ион-плазмалық азоттаудың негізгі принциптері, оның артықшылықтары мен шектеулері жан-жақты қарастырылып, титан қорытпаларының тозуға төзімділігі мен ұзақ қызмет ету мерзімін арттыратын оңтайлы өңдеу параметрлері анықталған. Жұмыста заманауи тәсілдер мен технологияны дамыту үрдістері талданған, бұл ең тиімді өңдеу режимдерін анықтауға және алынған қабаттардың сипаттамаларын жақсартуға мүмкіндік береді. Әсіресе өңдеу параметрлерінің – температура, қысым, газ қоспасының құрамы мен экспозиция уақыты – азотталған қабаттардың құрылымы мен қасиеттерінің қалыптасуына әсеріне ерекше назар аударылған. Зерттеулер көрсеткендей, дұрыс таңдалған өңдеу режимдері беткі қабаттың микрокаттылығын және тозуға төзімділігін арттырып қана қоймай, сонымен қатар үйкеліс коэффициентін төмендетуге мүмкіндік береді, бұл әсіресе жоғары механикалық жүктемелер жағдайында жұмыс істейтін бөлшектер үшін аса маңызды. Сондай-ақ ион-плазмалық азоттау титанды беткі қабаттық түрлендірудің басқа әдістерімен, мысалы, газдық азоттау, лазерлік беріктендіру және қатты жабындар жағу тәсілдерімен салыстырылды. Әдістің негізгі артықшылықтары ретінде азотталған қабаттың жоғары біртектілігі, бөлшектердің өлшемдерінің өзгерісінің минималды болуы және процестің экологиялық қауіпсіздігі анықталды. Алынған нәтижелер титан қорытпаларын өңдеудің өндірістік технологияларын оңтайландыру үшін қолданылуы мүмкін. Бұл олардың авиация-ғарыш, медицина және машина жасау салаларында сенімділік пен ұзақ қызмет ету мерзімі талап етілетін жағдайларда қолданылуын кеңейтуге мүмкіндік береді.

Түйін сөздер: плазма, жарқырау разряды, нитридті фаза, азот, температура, вакуум.

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Ионно-плазменное азотирование титановых сплавов: технологии, преимущества и ограничения

Для улучшения поверхностных свойств титана и его сплавов было разработано множество процессов модификации поверхности путем формирования покрытий, пленок или слоев. Одним из таких методов обработки является плазменное азотирование, которое наиболее широко используется для совершенствования поверхностных свойств титана и его сплавов. Диффузия азота в объемный материал создает непрерывный профиль твердости и, следовательно, обеспечивает оптимальную поддержку твердого поверхностного слоя. В данной работе исследуется технология ионно-плазменного азотирования для обработки титановых сплавов. Подробно рассмотрены основные принципы ионно-плазменного азотирования, его преимущества, ограничения, а также определены оптимальные параметры процесса, способствующие повышению износостойкости и долговечности титановых сплавов. Работа включает анализ современных подходов и тенденций в развитии технологии, что позволяет выявить наиболее эффективные режимы обработки и улучшить характеристики полученных слоев. Особое внимание уделено влиянию параметров обработки, таких как температура, давление, состав газовой смеси и время экспозиции, на формирование структуры и свойств азотированных слоев. Исследования показывают, что правильно подобранные режимы обработки позволяют не только увеличить микротвердость и износостойкость поверхности, но и снизить коэффициент трения, что особенно важно для деталей, работающих в условиях высоких механических нагрузок. Также проведено сравнение ионно-плазменного азотирования с другими методами поверхностной модификации титана, такими как газовое азотирование, лазерное упрочнение и нанесение твердых покрытий. Определены ключевые преимущества метода, среди которых высокая однородность азотированного слоя, минимальное изменение размеров деталей и экологическая безопасность процесса. Полученные результаты могут быть использованы для оптимизации промышленных технологий обработки титановых сплавов, что позволит расширить их применение в авиационно-космической, медицинской и машиностроительной отраслях, где требуется повышенная надежность и долговечность материалов.

Ключевые слова: плазма, тлеющий разряд, нитридная фаза, азот, температура, вакуум.

Introduction

Today, one of the key industrial challenges is to ensure the strength and wear resistance of various metals and alloys used in industries such as mechanical engineering, automotive and aerospace. This is especially true for titanium products that are subjected to significant mechanical stresses, such as in joint endoprostheses and dental structures. Constant friction, cyclic loads, and interaction with various materials, microcracks are formed as a result of fatigue failure of the material [1], which can lead to material failure. Despite titanium's high corrosion resistance and strength, its surface remains relatively soft, which increases the risk of wear, especially when in contact with metallic or ceramic elements. This shortens the service life of titanium products and reduces their reliability in long-term operation. To improve wear resistance and increase the service life of titanium alloy products, a promising solution is the use of chemical heat treatment (CHT) [2,3]. Among the most widespread methods of chemical heat treatment are cementation [4], nitrocementation [5], boriding [6] and nitriding. Each of these methods has its own features, advantages and limitations, which determines their field of application. Among the methods and technologies of CVD, ion-plasma nitriding stands out as the most promising technology due to a significant number of advantages. This process is based on saturation of the metal surface with nitrogen using a plasma discharge, which makes it possible to form high-quality nitride coatings with high hardness and wear resistance

Ion-plasma nitriding (IPN) is one of the promising technologies of surface treatment of materials, widely used to improve their mechanical and operational characteristics [7]. This technology is used to harden surfaces, improve corrosion resistance and increase wear resistance of metal products. IPN is widely used in various industries including mechanical engineering, metalworking, automotive and aerospace industries, energy and medicine. The technology is actively used to modify parts such as engine components, gears, shafts, cutting tools (drills, milling cutters, dies) and other products that require increased durability and efficiency under harsh operating conditions. Unlike traditional nitriding methods, such as gas or salt nitriding, IPN is carried out under low-pressure conditions in a vacuum chamber, where plasma activates nitrogen atoms and ensures their effective penetration into the surface layer of the material. This allows the creation of nitride layers of high density and homogeneity. In addition, IPN technology is characterized by environmental safety, as it does not require the use of toxic impurities and chemical reagents. Modern requirements to parts and tools include increased durability and stability under extreme conditions - high loads, aggressive media and significant temperature fluctuations. Ion-plasma nitriding not only meets these requirements, but also provides additional economic benefits by reducing wear and extending the service life of products

Ion plasma nitriding (IPN) technology continues to evolve rapidly, including the introduction of multi-cathode systems, the use of pulsed voltages, and combinations with other surface treatment methods such as oxidizing and coating. These improvements allow the method's capabilities to be significantly expanded and adapted to specific production requirements.

This paper discusses the basic principles of IPN operation, its advantages over other methods, existing limitations and ways of their elimination. It also presents an overview of current trends in the development of the technology and its application to the machining of complex metal shapes.

Ion-plasma nitriding process

The principle of operation of ion-plasma nitriding (IPN) is the formation of a nitride layer on the surface of a metal (steel, titanium, etc.). When voltage is applied between the anode (vacuum chamber body) and the cathode (the sample to be treated), a glow discharge is excited, which knocks electrons out of the atoms of the working gas, forming positively charged ions [8]. These ions bombard the metal surface, penetrating it and promoting the formation of nitride phase. As a result, the surface layer is modified, which increases its hardness, wear resistance and corrosion resistance. A detailed breakdown of the entire nitriding process is discussed below.

Fig. 1 shows the basic setup for ion-plasma nitriding. The vacuum chamber is pre-pumped to a pressure of 10^{-3} torr [9], which depends on the objectives and the type of metal to be processed. The required pressure is achieved by removing air and gas impurities using a pumping unit (Fig. 1, item 5). After the formation of plasma on the entire surface of the stand (Fig. 1, item 3) and the metal, the process of nitrogen ionization is activated. This process can be observed through the vacuum viewing window (Fig. 1, item 7). The working gas is exposed to a high-voltage electric field, which leads to its ionization, resulting in the formation of nitrogen ions (N^+) and neutral nitrogen atoms (Fig. 2). The nitrogen ions are accelerated in the electric field and collide with the metal surface, transferring their energy to it. This initiates processes such as the diffusion of nitrogen into the material and the formation of a nitride phase, which contributes to the hardness and wear resistance of the machined surface.

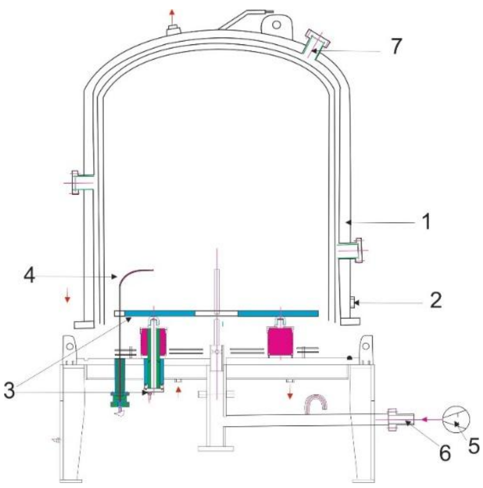

Figure 1 – Design of the IPN unit.
1-Vacuum chamber. 2-Anode. 3-Cathode (Support). 4-Temperature gauge of the vacuum chamber. 5-Pump unit. 6-Pressure relief plug. 7-Vacuum viewing window

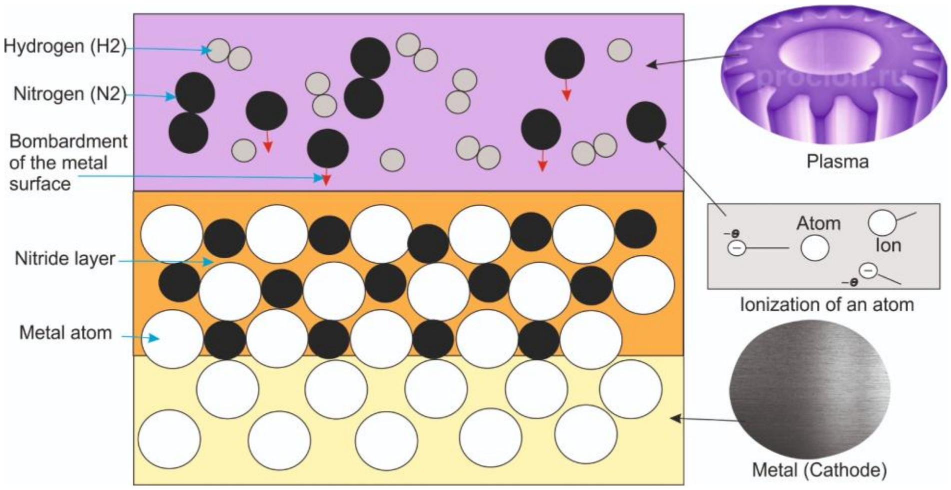

Figure 2 – The process of gas ion diffusion onto the metal surface.

When accelerated nitrogen ions and other plasma ions interact with the material surface, kinetic energy is transferred and converted into thermal energy. This process leads to heating of the treated sample. The temperature is controlled by a temperature sensor (Fig. 1, item 4) installed in the vacuum chamber. After reaching the set temperature, a timer is started, which fixes the duration of processing. At the end of the set time interval the temperature in the chamber is gradually reduced to room temperature. At the same time, a vacuum environment is maintained to prevent oxidation of the material surface. The voltage generator is only switched off after the temperature has stabilized to avoid temperature fluctuations that could adversely affect the material properties. After the cooling process is completed, inert gas is fed into the working chamber to prevent the treated surfaces from coming into contact with oxygen. The pressure in the chamber is released by opening the plug (Fig. 1, pos. 6), which ensures safe completion of the treatment cycle

Nitriding of complex metal shapes is a technically challenging process because it depends on many factors, including plasma physics, part geometry, and gas distribution characteristics [10]. In a vacuum chamber, the plasma can be unevenly distributed, especially in areas with recesses, threads, or complex topography. This leads to the fact that the internal surfaces of parts may not be saturated with nitrogen, which, in turn, contributes to the formation of "shadow zones" - areas with minimal ion density, where the nitriding process proceeds less efficiently [11].

Ion-plasma nitriding of complex metal shapes requires methods that ensure uniform treatment and efficient nitrogen saturation even in hard-to-reach places. To achieve this, various technological approaches and innovative solutions are used. The method of multi-cathode systems makes it possible to install several cathodes arranged around complex metal molds in a vacuum chamber. [12] The cathodes are placed in such a way as to minimize "shadow zones" and ensure uniform plasma exposure on all surfaces of the part. This ensures uniform plasma distribution and improves coatings in recesses, internal channels and complex topography. However, implementation of this technology requires additional cost and a skilled control approach. To provide better controllability of the plasma, pulse nitriding (pulse plasma) method is also used [13]. Which in turn applies voltages in the form of short pulses. This gives easy penetration of plasma into narrow channels and complex structures, minimizes overheating of parts with thin walls.

Favorable gas environments for diffusion to the surface of the metal product

Table 1 shows the different compositions of gas mixtures used in the ion-plasma nitriding process to achieve the specified properties of the treated material. The main active component in the mixtures is nitrogen (N_2), which provides saturation of the material surface with nitrogen atoms. Additionally, the gas mixtures may comprise hydrogen (H_2), argon (Ar), and hydrocarbons (e.g., methane - CH_4), which play an auxiliary role. Hydrogen (H_2) helps to remove oxides from the surface, improving the adhesion of the nitrided layers. Argon (Ar) stabilizes the plasma and regulates the ion energy, which affects the uniformity of the coating. Hydrocarbons (CH_4) are used to form combined coatings with increased wear resistance. The gas mixture is selected based on the requirements for thickness, hardness, corrosion resistance and other characteristics of the layer, as well as the properties of the treated material.

The role of each gas depends on the process objectives: nitrogen provides surface saturation, hydrogen cleans and stabilizes the process, inert gases improve surface activation, and hydrocarbons and oxygen complement the process, expanding the range of layer properties [14]. The optimum gas mixture is selected taking into account the material to be processed and the coating requirements.

The process of ionic nitriding in hydrogen-containing media (ammonia, nitrogen-hydrogen mixture) is well enough studied in domestic and foreign literature [15]. Hydrogen plays an important role in the process of ion-plasma nitriding, acting as an active component of the gas mixture. Its presence affects the kinetics of ionic interaction, the formation of the surface layer structure and the quality of the resulting coating. However, the presence of a large amount of hydrogen in the saturating medium causes surface brittleness (especially sharp edges) and base hardening, which under certain conditions leads to a decrease in the strength and durability of structural elements [16].

Table 1 – Compositions of gas mixtures for ion-plasma nitriding [14]

Gas combination	Assignment
NH_3	Provides plasma discharge stability, improving nitriding uniformity.
$N_2 + H_2$	A standard blend that provides uniform nitrogen saturation of the surface and improved adhesion properties.
$N_2 + Ar$	Used to increase plasma stability and improve processing quality.
$N_2 + H_2 + CH_4$	To create carbonitride layers with high wear resistance.
$N_2 + Ar + H_2$	For complex processes requiring high layer homogeneity

Influence of ion-plasma nitriding process parameters

The temperature of the ion-plasma nitriding process is a critical parameter that determines the quality and characteristics of the formed nitrided layer. Properties such as penetration depth, phase composition, surface roughness and corrosion resistance depend on carefully selected temperature conditions [17]. After ion plasma treatment, a significant increase in the microhardness of the metal surface is observed. However, it should be taken into account that excessively high microhardness can lead to surface embrittlement, which negatively affects its adhesion properties [18,19]. In addition, it can be observed, methods with high temperatures have higher microhardness. Therefore, for different metals and their alloys, an individual approach to the choice of temperature and processing conditions is required, which is due to the peculiarities of their structure and composition. The most widely used material for nitriding is steel, including its numerous grades. In the temperature range of 400-600 °C [17], which is typical for the processing of most types of steels, the diffusion rate of nitrogen atoms into the metal matrix increases significantly [17]. This leads to an increase in the thickness of the nitrided layer, which has a positive effect on its wear resistance. For titanium and its alloys, the process is usually carried out at higher temperatures - in the range of 700-900 °C, which is due to their specific physical and chemical properties [20]. With increasing temperature, the formation of ε-phase with increased corrosion resistance is possible. However, excessive heating can be accompanied by a decrease in the hardness of this phase, which in some cases limits its application. In addition, uncontrolled overheating can lead to deterioration of the properties of the base material, such as reduced ductility or internal stresses. Thus, temperature optimization is essential to balance layer thickness, hardness and corrosion resistance. A comprehensive approach, including temperature selection taking into account the properties of the treated material, makes it possible to achieve high efficiency of ion-plasma nitriding without compromising the mechanical characteristics of the base metal.

Vacuum also contributes to the stability and efficiency of the nitriding process [21]. Its application allows to remove impurities and oxide films from the surface of the material, contributing to a more uniform saturation of the metal with nitrogen. This is especially important in the processing of complex geometric shapes, where the uniformity of the coating significantly affects the mechanical properties of the product [22]. Maintaining a certain level of vacuum provides stability of the gas composition in the working chamber, which is important for the formation of a uniform and high-quality nitrided layer. According to [21], the use of vacuum allows to precisely regulate the parameters of the process of ion-plasma nitriding. The use of vacuum provides precise dosing of nitrogen and additional gas components such as hydrogen or argon, which has a positive effect on the intensity of diffusion processes and the thickness of the formed nitride layer. The vacuum environment reduces the probability of formation of pores, cracks, and undesirable inclusions in the surface layer, which contributes to its wear and corrosion resistance [21]. Despite the need for energy costs associated with vacuumization, the improvement in the quality of the formed layer and the increase in the service life of the machined parts compensate for these costs. Thus, vacuum is an indispensable element for achieving high performance in nitriding processes.

The choice of vacuum level depends on the purpose of the process. Medium vacuum (10^{-2}-10^{-3} Pa) is used to remove gaseous impurities such as oxygen and water vapor, which can negatively affect the process. It provides the necessary cleanliness of the working chamber before supplying the working gas (nitrogen or a mixture with other gases). The operating vacuum (50-500 Pa) is set after the supply of the operating gas and its value is determined by the process parameters, material type and product geometry. High vacuum (10^{-5}-10^{-7} Pa) is used in specialized installations for maximum complete removal of contaminants, especially when working with high-alloy steels and titanium [8].

The next voltage parameter affects the formation of plasma and regulation of nitrogen ionization intensity. Under the action of high voltage between the anode and cathode, an electric field accelerating ions and electrons is generated, which contributes to the initiation and maintenance of the plasma state in the working chamber [18]. In addition, the voltage affects the energy distribution in the system, which directly affects the efficiency of the nitriding process. When the voltage is increased, an increase in the energy of nitrogen ions is observed, which promotes deeper penetration of nitrogen atoms into the metal and improves the properties of the nitrided layer, such as wear resistance and strength. However, excessive stress can lead to overheating of the material surface, which in turn can damage the material, especially with prolonged exposure, requiring precise control of the process parameters. In addition, stress affects the morphology and structure of the nitrided layer. At high stress, accelerated nitrogen ions activate the interaction processes with the material, leading to the formation of denser and more wear-resistant layers. However, excessive stress can promote the formation of unfavorable phases such as the ε-phase, which improves corrosion resistance but can reduce the hardness of the coating. Thus, the optimal choice of voltage for ion-plasma nitriding should take into account the specifics of the material, the type of product, the requirements for the properties of the nitrided layer, and other process parameters [8]. It is important to ensure the voltage balance in order to achieve the desired mechanical and physicochemical characteristics, minimizing the risk of material damage and the occurrence of undesirable phases.

The time of ion-plasma nitriding is an important parameter determining the thickness, phase composition, and mechanical characteristics of the formed nitrided layer [23]. Increasing the treatment time promotes the growth of the diffusion layer thickness due to the intensification of the diffusion of nitrogen atoms into the metal matrix, although over time the process slows down due to the decrease in the concentration gradient. The duration of exposure affects the phase composition of the layer: at early stages, the γ'-nitride phase predominates, while longer nitriding promotes the formation of ε-phase with increased corrosion resistance [24]. However, excessive processing time can lead to the formation of saturated nitrides (e.g., TiN, CrN), increased surface roughness and reduced ductility of the base material due to internal stresses and microcracks. In addition, excessive processing time increases energy consumption, reducing cost effectiveness. Thus, to ensure a uniform nitrided layer with optimum mechanical properties such as hardness, wear resistance and corrosion resistance, the process time must be carefully optimized depending on the material, product geometry and operational requirements. Table 2 presents a comparative analysis of key parameters of the ion-plasma nitriding process and their influence on the characteristics of the coatings. The parameters included - temperature, pressure, voltage, gas mixture composition and treatment duration - are critical for the formation of nitride layers with optimum mechanical properties. Analysis of experimental data shows that for each of these parameters, there is an optimal range of values that provides a balance between hardness, wear resistance, corrosion resistance, and minimization of undesirable effects [25] such as overheating or non-uniformity of the coating. This information will be useful for further research in the field of surface treatment of titanium and its alloys.

Table 2 – Influence of ion-plasma nitriding parameters on coating characteristics

Process parameter	Effect on coverage	Mechanism of influence	Analysis of experimental data	Conclusions
Temperature	Increasing hardness and wear resistance, formation of nitride phases	Diffusion of nitrogen atoms into the matrix, formation of ε- and γ'-phases	Optimum temperature for titanium: 700-900 °C, for steels: 400-600 °C. Excessive temperature can lead to a decrease in ductility	Temperature conditions should be selected individually for each material
Chamber pressure	Influence on the uniformity of nitrogen saturation	Removing impurities, improving plasma stability	Medium vacuum (10^{-2}-10^{-3} Pa) is required for removing impurities, working vacuum (50-500 Pa) is required for processing	Vacuum optimization reduces the probability of defect formation
Voltage	Determines ion energy, depth of diffusion	Acceleration of ions in the electric field	Higher stress increases the hardness of the coating, but excessive stress can cause overheating	Balance between energy and the possibility of surface overheating
Gas mixture composition	Effect on structure and adhesion of coatings	Regulation of interaction and ionization kinetics	Mixtures of $N_2 + H_2$, $N_2 + Ar$, $N_2 + CH_4$ allow to obtain coatings with different properties	Selection of the mix depends on the requirements of the coating
Treatment duration	Increase in the thickness of the nitride layer	Continuous diffusion of nitrogen,	The coating thickness increases with increasing time, but overheating and roughness may occur	Optimized processing time reduces energy costs and improves coating properties

Conclusion

Ion-plasma nitriding is a highly efficient method of chemical and thermal treatment of metal surfaces, which allows to significantly improve the performance characteristics of products. By using plasma in a vacuum chamber, a deep and uniform saturation of the surface with nitrogen is achieved, resulting in the formation of a strong nitride layer. This layer provides a significant increase in hardness, wear resistance, fatigue strength and corrosion resistance of materials without changing their dimensions and structure. The main advantage of the method is its ability to provide precise control of process parameters such as temperature, pressure, gas composition and ion current density. This allows the process to be customized to meet the specific machining requirements of different alloys and steels. In addition, ion-plasma nitriding is environmentally friendly and cost-effective compared to traditional nitriding methods such as gas or liquid nitriding. The technology is widely used in mechanical engineering, aerospace, automotive and power generation. It is used to treat parts subject to high mechanical stress and friction, such as gears, shafts, dies and cutting tools. Due to the possibility of localized machining, this method is also used to modify surfaces of complex geometric shapes. The development of ion-plasma nitriding is aimed at improving plasma sources, reducing energy consumption and expanding the range of processed materials. Studies on combining ion-plasma nitriding with other methods of surface hardening, such as nitrocementation and ion alloying, are underway, which opens new horizons in the creation of high-performance coatings with specified properties. Thus, ion-plasma nitriding is the most important technology of modern materials science, providing reliability, durability and high performance of products in a wide range of industries. Its further development and integration with innovative technologies will contribute to the creation of new materials and structures that meet the growing requirements for strength and durability under conditions of intensive operation.

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