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Nitriding in a cyclically switched glow discharge

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Abstract

The paper shows the prospects of nitriding in a cyclically switched discharge, the classification of nitriding processes in a glow discharge according to the criteria of the characteristics of the power source is given. A comparison of the advantages and disadvantages of nitriding processes with constant and cyclically switched discharge is given. From a theoretical point of view, the process of nitriding in a cyclically switched glow discharge is considered based on the concept of an energy model. In accordance with this model, tasks for further theoretical and experimental research are formulated. It is shown that the process of surface modification in a cyclically switched by: frequency, period and pulse shape. The implementation of the process of adjusting the switching frequency, pitch - the ratio of the cycle period to the duration of the signal, and the shape of the signal itself opens up wide opportunities to significantly influence the results of surface treatment.

The influence of the shape of the discharge power signal on the kinetics of the nitriding process and its results opens up wide opportunities for studying the process itself. The presence of surges at the beginning and at the end of the cycles can, in principle, significantly affect both the nature of the nitriding process itself and the structure and phase composition of the modified surface layer, since short-term and sufficiently powerful voltage surges should lead to intensive surface sputtering. The destruction of the monolayer of nitrides, which has just formed on the surface, will contribute to the increase of the depth of the nitrided layer due to the diffusion of nitrogen particles, as well as to a certain extent leveling off the blocking effect of the surface nitride layers.

Keywords: nitriding, glow discharge, cyclic switching, discharge power, waveform, nitride layer depth

Introduction

Nitriding in a glow discharge with constant or conditionally constant power is traditionally used as one of the effective methods of surface modification of metals. For this method of strengthening metal surfaces and alloys, both the main theoretical provisions and practical recommendations for its application have been established [1-3].

Nitriding in a cyclically switched (intermittent) glow discharge (CSD) opens up fundamentally new possibilities both theoretically and technologically. First of all, this concerns a significant simplification of the principles of the formation of the cage, as well as the danger of the glow discharge turning into an arc. Despite the obvious new possibilities opened up by the application of processes in a cyclically switched glow discharge, theoretically this process has hardly been developed, which, of course, does not contribute to the use of all its potential. A separate aspect in the theory of the nitriding process in a cyclically switched discharge is its interpretation from the energy point of view, since in the end all elementary sub-processes are fundamentally regulated by energy prerequisites. By creating certain energy parameters of the process, it is possible to stimulate or, on the contrary, inhibition of the main competing components: formation of nitrides, diffusion of nitrogen into the depth of the surface, sputtering of surface layers. The characteristics of tribological systems on the modified surface of metals depend on the ratio of these components. In addition, the question of the adequacy of the evaluation of the results of surface modification to the practical performance indicators of the modified products is essential. The process is absolutely environmentally friendly. The principle of modification of the metal surface, which is used in the researched process, provides the most favorable economic prerequisites compared to other



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Classification of nitriding processes

The general systematization of surface modification processes using glow discharge qualifies them as vacuum-diffusion gas discharge [2, 4, 5]. Most of the theoretical studies of the process refer to one of its possible variants, where the power source is continuously loaded on the discharge chamber. However, in principle, other power options are also possible, and in this case, certain technological advantages are achieved.



Fig. 1. Classification of nitriding processes in glow discharge according to the criterion of power source characteristics

Consideration of nitriding in a glow discharge precisely according to the criterion of continuity of power leads to the scheme shown in Fig. 1. In the vast majority of cases, nitriding installations in glow discharge were formed according to the scheme of direct polarity (conventional name), in which the part is the cathode. In theory, the doctrine prevailed, according to which nitriding of parts to which the positive pole of the power source is attached (reverse polarity) is impossible [6]. However, later a number of studies were conducted, including experimental ones, which proved that nitriding is possible even with such a feeding scheme. At the same time, it is only important to provide a method other than cathodic bombardment to heat the parts to the temperature regulated by the technological regime. This version of the process is especially effective when using an arc discharge, when the current density is significantly higher compared to glow discharge [7]. In the future, the nitriding method with reverse polarity, in the case of sufficient theoretical work, can be used as one of the really

possible options, since it creates conditions for a relatively low energy of the incident flow in the area of nitride formation energies. Thus, the low-energy spectrum of the incident flow may contribute to the growth of the activity of nitride formation while reducing the intensity of their sputtering [8].

In chronological order, technologies with continuous power supply were introduced first. Such systems are fundamentally simpler, and, most importantly, ensure the transfer of a greater amount of energy to the falling flow and, accordingly, to the processed parts. In the complex, this contributes to a greater intensity of modification and a shorter duration. Better stability of the discharge is observed when using ripple filters, but this complicates power supply units and their cost, in addition, electricity costs increase [2].

In AC power supply units, there are options for completely or partially cutting off one of the phases (as a rule, the positive one, if a process with direct polarity is used). Without such a cut-off, the heating of the anode, as a rule, the walls of the chamber increases and the intensity of nitriding decreases. However, the latter can lead to a softer regime of nitriding, in accordance with better indicators of plasticity of the modified layer. A certain reduction of the negative influence of the positive phase on the nitriding process can be achieved by the asymmetry of the voltage shape due to the displacement of its average value in the negative zone. In principle, the shape of the voltage can be sinusoidal, rectangular or arbitrary up to its programmed change. It is obvious that the use of a rectangular voltage form with other constant parameters provides a greater energy saturation of the incident flow, accordingly, the regime is more rigid, but the processing time is shorter. The simplest is the option of using the transformed voltage of the industrial frequency, because it eliminates the need to use rectifiers. High-frequency current feeding is somewhat similar in principle (excluding the effect of the positive phase) to nitriding with intermittent feeding.

Nitriding with constant feeding has a significant technological disadvantage. It consists in the increased complexity of garden formation. This is primarily due to the fact that in all places where there are gaps larger than 0.5 mm, local burning of the discharge occurs, but already in the arc mode. This leads to local overheating of the parts, and in some cases, to the impossibility of conducting the process, because an arc occurs when trying to increase the voltage in such places. The need to take into account the mentioned circumstance complicates the construction of hangers or tables that would ensure a tight base of parts on them. However, even when the condition of a tight base of the part on the table is met, the presence of chamfers on the supporting surface of the parts also causes a certain problem, since it together with the supporting surface of the table forms a wedge-shaped gap. Another drawback, especially in comparison with gas furnace nitriding, there is a low efficiency of modification of holes, especially of small diameter, with their relatively long length. From the theory of discharge with a hollow cathode, it is known that the field strength in the hole is practically zero at a depth of more than two diameters [2]. The necessary results of nitriding of the holes can be achieved due to a significant increase in the duration of the treatment, but in such a setting, all other advantages of nitriding in the glow discharge are negated, especially in terms of duration, since in this case the process follows the scheme of ordinary furnace gas nitriding.

Nitrogenation in the CSD

In recent years, nitriding technologies in glow discharge with intermittent power supply - cyclically switched discharge (CSD) - have been used in increasingly noticeable volumes. The main principle of the formation of the CSD is that the duration of the signal in the cycle should not be longer than the time of the transition of the glow discharge to the arc, and the pause should not be less than the extinguishing of the arc discharge, if it even occurred. Under such conditions, there is practically no need for automatic arc discharge cutting devices, or they can be used according to a scheme with an extremely simplified algorithm. The cage can be formed in compliance with the requirements, which are no more complicated than in the case of ordinary furnace nitriding. In other words, if we ignore the issue of uneven surface temperature distribution caused by mutual heating from the adjacent part, then in the garden they can be placed practically in a heap or use any supporting surfaces, hooks for hanging, etc., At the same time, existing local surface exceptions, gaps, etc., play almost no negative role. It has been experimentally established that, using CSD, it is possible to nitride small-diameter holes with a relatively significant depth [9, 10]. However, since the energy transfer to the falling flow takes place only in part of the cycle, the efficiency of nitriding is lower, and in some cases the actual nitriding phase is two or even more times longer than with continuous nitriding [11]. By the way, with regard to the terminology used in this work, it should be noted that it is fundamentally incorrect, since we deal with voltage pulsations all the time even during normal nitriding, especially in cases where filters are not used.

Intermittent power supply with the help of CSD is characterized in terms of classification by several characteristics: frequency or period and pulse shape. It would be optimal to be able to adjust the switching frequency, since it, together with the ability to change the frequency - the ratio of the cycle period to the duration of the signal - will have the most significant effect on the processing results. The option using a high-frequency field allows the use of standard power generators (provided that the positive phase is cut off), although there are problems with the shielding of the installation elements to prevent the occurrence of radio interference. If sources are used that allow adjusting the gap, then in principle it is possible to implement both continuous and intermittent power supply options in one installation. Such versatility allows you to optimally use the potential possibilities of the process, because in the presence of complications related to the shape of the parts (local exceptions, holes, etc.), you can use CSD, while the period of the actual nitriding phase will increase. If the shape of the part is not

complicated. then it is advisable to switch to a more productive continuous power supply method. A similar choice can be made when there is a need to apply less rigid technological regimes. With regard to the shape of the pulse, this question should first of all be considered as an object of theoretical studies of the formation of the energy spectrum of the falling flow under alternating voltage, both in the phase of voltage increase and its decrease. A certain role, perhaps in this sense, can be played by tension fronts. parts related to the shape (local exceptions, holes, etc.) can be used by CSD, while the period of the actual nitriding phase will increase. If the shape of the part is not complicated, then it is advisable to switch to a more productive continuous power supply method. A similar choice can be made when there is a need to apply less rigid technological regimes. With regard to the shape of the pulse, this question should first of all be considered as an object of theoretical studies of the formation of the energy spectrum of the falling flow under alternating voltage, both in the phase of voltage increase and its decrease. A certain role, perhaps in this sense, can be played by tension fronts. parts related to the shape (local exceptions, holes, etc.) can be used by CSD, while the period of the actual nitriding phase will increase. If the shape of the part is not complicated, then it is advisable to switch to a more productive continuous power supply method. A similar choice can be made when there is a need to apply less rigid technological regimes. With regard to the shape of the pulse, this question should first of all be considered as an object of theoretical studies of the formation of the energy spectrum of the falling flow under alternating voltage, both in the phase of voltage increase and its decrease. A certain role, perhaps in this sense, can be played by tension fronts. A similar choice can be made when there is a need to apply less rigid technological regimes. With regard to the shape of the pulse, this question should first of all be considered as an object of theoretical studies of the formation of the energy spectrum of the falling flow under alternating voltage, both in the phase of voltage increase and its decrease. A certain role,

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Theoretically, the process of nitriding in a cyclically switched glow discharge (CSGD) is supposed to be considered based on the concept of the energy model [2, 12]. The main differences and additions to previously performed theoretical studies are reduced to the following:

1) the kinetics of the movement of the particles of the incident current in the near-cathode zone will have a feature related to the fact that at the moment of pause (absence of voltage) the charged particles will no longer move under the influence of the electric field strength, but by inertia tangential to their previous one at the moment of removal potential of the trajectory field;

2) a change in the kinetic dependences, including the values of the parameters of the motion of the mediated particle, will affect the probabilistic characteristics of the falling particles reaching the cathode, as well as the angular parameters of their collision with the surface. In turn, the angular characteristics will change the conditions of energy transfer from the particles of the incident flow to the surface components;

3) removing the field tension at certain moments of particle movement will stop their accumulation of kinetic energy, which will affect the distribution of particles by energy levels;

4) therefore, the energy spectra of the incident flow will change, depending not only on the traditional parameters of the technological regime, but also on the characteristics of the specified CSD;

5) since all the main indicators of the energy model - relative energy factors primarily depend on the shape of the energy spectra, the change of the latter requires additional research;

6) the formation of the structure of the modified layer also changes, the main dependences of the processes of phase formation during CSD require separate studies;

7) the most significant consequence of the processes of formation of tribotechnical systems is the study of the influence of their structure on the wear resistance of modified layers. The practical result is the establishment of regularities of technological process control depending on the necessary processing results, formed on the basis of the requirements of the conditions of the subsequent operation of the objects;

8) for the practical application of the positive nitriding factor of small-diameter holes when using CSD, it is necessary to develop a theory of changes in the concentration of diffusants in relatively long holes;

9) since the energy balance of plants operating with the use of CSD can significantly differ from the similar indicator of plants with continuous power, this issue requires a separate study;

10) when applying CSD, conditions are created for controlling the intensity of phase formation by changing the rigidity of the technological regime, therefore, the issue of nitriding in CSD of nitrogen-active metals is of particular practical interest;

11) it is also necessary to investigate the possible influence on the NCSD process of the shape of the voltage change and the deviation of this shape from the ideal;

12) an important role will be played by the processes in the internal local exceptions of the surface, since it will be necessary to compare the behavior of such surface elements in the process of nitriding under different power options;

The noted problems form the basis of a new scientific and technical task - nitriding of metals in a cyclically switched discharge. The practical implementation of the research results is primarily aimed at using the positive properties of nitriding processes with intermittent discharge discharge.

Hardware implementation of nitriding in CSD

According to the data given in works [9-10], the introduction of the cyclic switching mode during nitriding in the glow discharge opens up additional opportunities to improve the efficiency and quality of the specified technology, primarily:

- the possibility of nitriding parts of a complex shape is expanded (the presence of deep and narrow grooves, small-diameter holes, long holes, etc.);

- the risk of local damage to modified surfaces is reduced by reducing the probability of arc discharges;

- the permissible range of adjustment of parameters important for optimizing the technological process, such as the pressure in the discharge chamber and the temperature of the surface of the parts, is expanded;

- the control of preventing overheating of the surface is significantly simplified due to the release of additional discharge energy.

The development of the experimental layout of the device for the implementation of cyclic switching of the gas discharge was based on the following prerequisites:

- maximum compatibility with existing electrical and electronic equipment;

- use of available element base;

- the possibility to provide a sufficiently wide range of switching parameters for the purpose of further optimization of modes and their comparative analysis;

- reliable equipment protection against overloads and abnormal situations.

The justification of the switching parameters is primarily based on the following requirements for the pulse current source:

- the shape of the pulse should be rectangular, which would ensure a jump from the zero level to the desired zone of anomalous glow discharge;

- the duration of the pulse should be shorter than the arc development time (approximately less than 100 µs), while the formation of the arc is disturbed; if necessary, the current can be interrupted during any pulse;

- the pause that follows each pulse should be short enough to ensure easy ignition of the discharge under the action of the next pulse, i.e. be less than a few milliseconds;

- the ratio of the duration of the pulse and the pause should vary widely to effectively control the heating of the parts.

Typical values of the pulse duration, which are recommended in [10], are in the range from 20 to 100 μ s, while the pause duration can vary from 20 to 200 μ s. It should be noted that the processes of ignition and extinguishing of the discharge are characterized by significant inertia, and this circumstance imposes certain restrictions on the choice of time parameters of switching. Moreover, the inertia of gas discharge processes may depend on the geometry of the discharge space and its dimensions, which in real conditions can vary in a very wide range. The conclusion follows that it would be imprudent to unconditionally transfer the recommendations [10, 13], which were developed on an experimental installation with a chamber with a diameter of 400 mm and a height of 600 mm, to the case of much larger installations. The same applies to the results, obtained in the process of physical research of miniature gas discharge devices (the so-called laboratory discharge). However, it was decided at this stage of development to provide a sufficiently wide range of current switching parameters in order to refine it after conducting a series of preliminary experiments. Namely, we specify the ranges of variation of the frequency f of pulse tracking from one to ten kilohertz, the filling factor of the period from zero to one:

$$\eta = t_n / T = 0...1$$

where T is the pulse tracking period T = 1/f,

 t_n is the duration of the pulse (duration of the active part of the period).

In order to save time and costs, it was decided to use the power supply unit of the existing plants for nitriding in the glow discharge and the corresponding control devices (CSD - nitriding process controller), as well as current and discharge voltage sensors. To implement the pulse mode of the installation, the power supply unit is additionally equipped with a T-shaped RC - smoothing filter and a specially developed intermittent mode controller (IMC), which includes a power electronic key EC with a control and protection device.

For the construction of the power switch, a powerful transistor of the MOSFET structure was chosen, which is characterized by the following advantages, first of all - in comparison with bipolar transistors:

- low power of consumption in management circles;

- good characteristics when working in parallel, which makes it relatively easy to increase the power of the key (up to certain limits).

At the same time, as with the use of bipolar transistors, in this case there is an urgent problem of protection against current overloads, which sharply reduce the reliability of the key.

The device for implementing cyclic switching of a gas discharge is described in detail in [14].

The practical implementation of the device on a nitriding installation in a smoldering cyclic-commutated

discharge confirmed its operability in the conditions of real technological processes. At the same time, all the planned prerequisites mentioned above have been achieved, which opens the way to the experimental use of cyclically switched discharge for surface modification of metal alloys on a fundamentally new basis. The application of this method allows solving a number of technological problems, the most important of which is the modification of parts of a complex shape, with holes and depressions of small transverse and large dimensions. It was possible to effectively nitride such parts only with the use of furnace nitriding in ammonia gas environments, that is, a process that is unacceptable in modern conditions not only in view of its economic indicators, but also, first of all, from the point of view of environmental safety. In addition, the probability of surface damage caused by the accidental transition of a glow discharge to an arc, which is often observed when using continuous power supply of the discharge chamber, is significantly reduced.

Practical implementation of nitriding in CSD

The presence in the installation scheme of the block of cyclic switching of the discharge does not exclude the possibility of using the equipment in the constant discharge mode, providing for the possibility of mobile variation of the modes: constant and switched.

In AC power supply units, it is possible to completely or partially cut off one of the power lines (as a rule, the positive one, if a process with direct polarity is used). The absence of such a cut-off helps to increase the heating of the anode, which leads to a decrease in the intensity of the process. However, such a phenomenon can lead to softening of the nitriding process itself, obtaining better indicators of plasticity of the modified layer. A certain reduction of the negative influence of the positive phase on the nitriding process can be achieved by the asymmetry of the voltage shape due to the displacement of its average value in the negative zone. In principle, the shape of the voltage can be sinusoidal, rectangular or arbitrary up to its programmed change. The influence of the signal shape on the process of formation of the modified surface layer requires a separate study. Obviously, that the use of a rectangular form of voltage with other constant parameters ensures a greater energy saturation of the incident flow, correspondingly, a greater rigidity of the regime, with a shorter duration of processing. The simplest is the option of using a transformed industrial frequency voltage, which is somewhat similar in principle (excluding the effect of the positive phase) to nitriding with intermittent power supply.

The use of cyclically switched power discharge of the discharge chamber, which is carried out by current in the form of an intermittent signal, has a number of significant advantages:

- the possibility of forming such a CCR, in which the duration of the signal in the cycle does not exceed the time of transition of the glow discharge into the arc. The duration of the pause is not less than the extinguishing time of the arc discharge, in the event of its occurrence, which negates the need for automatic arc discharge cutoff devices, or they can be used according to a scheme with an extremely simplified algorithm;



Fig. 2. The shape of the change in the discharge current with a cyclically switched discharge of a rectangular shape with a slit $\gamma = 2$.

- the process of forming the cage is significantly simplified, since the need to comply with the requirements for gaps and deep holes practically disappears;

- there is an opportunity to nitride holes of small diameter with a relatively significant depth;

It should be noted as a disadvantage when using the CSD that the energy transfer to the falling flow takes place only in that part of the cycle where the signal is active and the efficiency of the process is lower, and in some cases the nitriding phase is two or even more times longer than with a continuous discharge. The nature of the change in current during a cyclically switched discharge is shown in Fig. 2, voltages - in fig. 3.

However, in general, the surface modification process in a cyclically switched discharge opens up new possibilities associated with variants of the CCR itself, which is characterized by: frequency, period and pulse shape. The implementation of the process of adjusting the switching frequency, pitch - the ratio of the cycle period to the duration of the signal, and the shape of the signal itself opens up wide opportunities to significantly influence the results of surface treatment.



Fig. 3. The shape of the discharge voltage change with a CSD of a rectangular shape with a slit $\gamma=2$.

The influence of the shape of the discharge power signal on the kinetics of the nitriding process and its results opens up wide opportunities for studying the process itself. The presence of surges at the beginning and at the end of the cycles can, in principle, significantly affect both the nature of the nitriding process itself and the structure and phase composition of the modified surface layer, since short-term and sufficiently powerful voltage surges should lead to intensive surface sputtering. The destruction of the monolayer of nitrides, which has just formed on the surface, will contribute to the increase of the depth of the nitrided layer due to the diffusion of nitrogen particles, as well as to a certain extent leveling off the blocking effect of the surface nitride layers.

Conclusions

The use of cyclically switched discharge nitriding in glow discharge technology allows to obtain adjustable and even predictable processes of surface modification of metals and alloys, formation of surface layers with specified properties, especially for parts of complex configuration.

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Стечишин М.С., Диха О.В., Олександренко В.П., Стечишина Н.М. Азотування в циклічнокомутованому тліючому розряді

В роботі показані перспективи азотування в циклічно-комутованому розряді, наведена класифікація процесів азотування в тліючому розряді за критерієм характеристик джерела живлення. Приводиться порівняння переваг і недоліків процесів азотування з постійним і циклічно-комутованим розрядом. В теоретичному плані процес азотування в циклічно-комутованому тліючому розряді розглядається виходячи з концепції енергетичної моделі. У відповідності до цієї моделі сформульовані завдання для проведення подальших теоретичних та експериментальних досліджень. Показано, що процес модифікації поверхні в циклічно комутованому розряді відкриває нові можливості, пов'язані з варіантами самого ЦКР, який характеризується: частотою, періодом та формою імпульсу. Реалізація процесу регулювання частоти комутації, шпаруватості – відношення періоду циклу до тривалості сигналу, та форми самого сигналу відкриває широкі можливості суттєво впливати на результати обробки поверхні.

Вплив форми сигналу живлення розряду на кінетику процесу азотування та його результати відкриває широкі можливості для дослідження самого процесу. Наявність сплесків на початку та в кінці циклів в принципі може суттєво впливати як на характер самого процесу азотування, так і на структуру та фазовий склад модифікованого поверхневого шару, оскільки короткочасні та достатньо потужні сплески напруги повинні призводити до інтенсивного розпорошення поверхні. Руйнування моношару нітридів, які щойно утворились на поверхні, сприятиме збільшенню глибини азотованого шару за рахунок дифузії часток азоту, а також нівелюватиме до певної міри блокуючий ефект поверхневих нітридних шарів.

Ключьові слова: азотування, тліючий розряд, циклічна комутація, живлення розряду, форма сигналу, глибина нітридного шару