
PLASMA MODIFICATION OF THE SURFACE OF CONSTRUCTIONAL MATERIALS

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Methods applied in mechanical engineering for the plasma modification of metal surfaces have been discussed with a special emphasis being put on the advantages of promising ion-plasma technologies. The results of our researches devoted to the volume arc discharge supplied by a plasma source of electrons fabricated as a hollow cathode with gas-magnetron heating have been presented. The advantages and shortcomings of energy efficient plasma-diffusion technologies for nitriding in glowing and volume arc discharges, as well as the prospects of their development, have been analyzed.

1. Introduction

One of the most important operations in hi-tech technologies of modern mechanical engineering is the thermochemical treatment (TCT) of details and tools, which improves major properties of constructional materials, such as wear resistance, corrosion stability, and fatigue and heat resistances. The main origin of low lifetime for the majority of details is the wear of their working surfaces. An enhancement of the wear resistance of materials implies the application of surface modification methods aimed at implementing the required changes in the physical and chemical properties of the surface layer.

Nitriding, one of the TCT kinds for metals, consists in saturating the surface layers of metal products by nitrogen. Such layers are characterized by enhanced hardness, wear and fatigue resistances, and corrosion stability in various media [1]. In the course of standard nitriding, products are heated up in furnaces in the ammonia atmosphere at a temperature of 500–600 °C; in this case, ammonia partially dissociates into atomic hydrogen and nitrogen. The latter is adsorbed onto the metal surface and diffuses into the crystal lattice, sequentially forming different nitrogenous phases in the metal bulk. It is these phases that govern the special properties of the modified metal surface.

Among the modern methods of surface modification, most promising are the gas-discharge ion-plasma TCT technologies, which enable coverages with preset properties to be formed. They are based on the

interaction between the metal surface and ions, as well as other particles, obtained in a low-temperature plasma with the given composition [2]. From the viewpoint of general TCT methods, plasma in this case is a technological saturating environment, which affects the surface of the treated metal. For instance, a gas discharge in the rarefied atmosphere of nitrogen (or its mixtures with other gases) allows ions and atomic particles to be readily obtained as a result of elementary processes running in the discharge plasma. By applying a negative – with respect to the plasma – potential to a treated product or using it as a glowing discharge cathode, the product surface is subjected to ion bombardment, destined for the effective cleaning of the product surface and its heating to a required temperature. Therefore, in contrast to standard TCT methods, plasma – besides fulfilling the functions of a technological atmosphere – is also an agent of active energy influence upon the surface to be modified.

From the viewpoint of energy efficiency, the TCT of materials provides the double effect. First, the general result of their application is a substantial reduction of the resource and, hence, power consumptions in technological processes, where constructional materials subjected to the TCT are manufactured and applied. Second, the ion-plasma methods are more beneficial energetically in comparison with the traditional ones that use furnaces, at least owing to the exclusion of a massive furnace equipment from the technological process. As a result of all reasons mentioned above, the ion-plasma technologies aimed at the creation of functional and protective coatings are intensively developed throughout the whole world.

In this work, the efficiency of the processes of nitriding of metal surfaces in a volume arc discharge and a glowing discharge has been experimentally studied.

2. Experimental Installation

2.1. Arc discharge

The simulation of the nitriding process in a non-self-sustained volume arc discharge was carried out in a

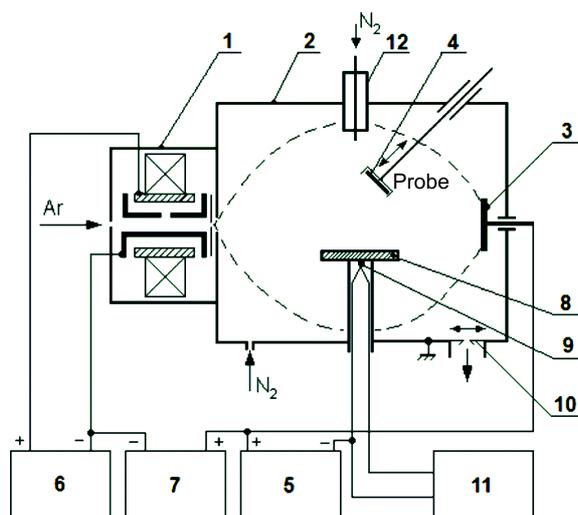


Fig. 1. Scheme of a plasma generator for implementing the process of nitriding in volume plasma: 1 – cathode assembly (gas magnetron), 2 – vacuum chamber, 3 – anode, 4 – probe, 5 – bias voltage power unit, 6 – gas magnetron power unit, 7 – volume arc discharge power unit, 8 – specimen stage, 9 – thermocouple, 10 – control diaphragm, 11 – temperature measurement unit, 12 – nitrogen inlet device

cylindrical 100-l vacuum chamber. The cathode and anode assemblies were located at the lateral covers of the latter (Fig. 1).

The cathode assembly was fabricated on the basis of a hollow cathode with gas-magnetron heating; it fulfilled the functions of the plasma emitter of electrons [2]. The body of the cathode itself was fabricated of molybdenum in the form of a hollow cylinder containing a tantalum insert. The working part length of the cathode was 40 mm, its internal diameter was 3 mm. The hollow cathode was connected with the working volume of the chamber through a tungsten diaphragm 2 mm in diameter. An auxiliary discharge to heat up the hollow cathode and effectively ignite the main discharge was obtained making use of a gas magnetron [2].

After applying the voltage from a power unit to the gas magnetron electrode system (the hollow cathode and the cylindrical anode) and letting a ballast gas (Ar) into the hollow cathode, where, owing to the resistance of the diaphragm located between the cathode assembly and the chamber, a region with an increased pressure emerges, electrons, being subjected to the action of crossed electric and magnetic fields, start the rotational motion around the cathode. This gives rise to the growth of the probability for electrons to collide with atoms and, hence, results in both the gas ionization and the

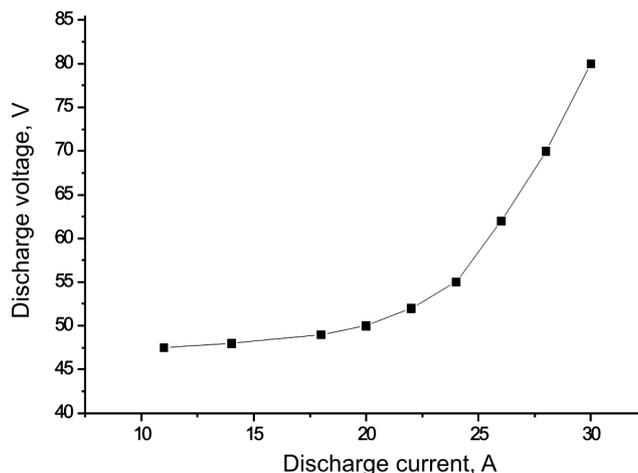


Fig. 2. Current-voltage characteristic of volume arc discharge. The consumption of a plasma-generating gas (argon) is $0.15 \text{ cm}^3/\text{s}$

discharge ignition in the gas magnetron. The increase of the discharge power in the magnetron up to 500 W is accompanied by the growth of the hollow cathode temperature to a level, at which thermionic emission emerges. Plasma generated in this discharge penetrates into the hollow cathode through a lateral aperture. Being subjected to the action of an electric field between the hollow cathode and the anode, electrons emitted from the plasma around the hollow cathode are accelerated to ionize the saturated atmosphere gas in the vacuum chamber. As a result, a volume arc discharge becomes ignited between the hollow cathode and the ring anode 245 mm in diameter and located in the chamber at a distance of 650 mm from the cathode. The discharge input power can be controlled by varying the power unit reactance.

The arc discharge was maintained in the environment of nitrogen or a nitrogen-argon mixture at a pressure of 0.1–1.5 Pa in the range of working currents 5–30 A (see the current-voltage characteristics in Fig. 2). According to the estimations made, the electron concentration was $10^{10} - 10^{11} \text{ cm}^{-3}$.

One of the most important experimental parameters was the ionic current density which can be taken off from plasma. It was determined with the help of a unilateral 10-cm^2 flat probe, the negative potential of which was down to -300 V . The probe was movable, which enabled an opportunity to measure the dependence of the ionic current density on the radial coordinate (see Fig. 3). The distributions depicted in this figure were obtained in argon plasma and at a fixed argon flow rate of $0.15 \text{ cm}^3/\text{s}$. The pressure was varied by controlling the

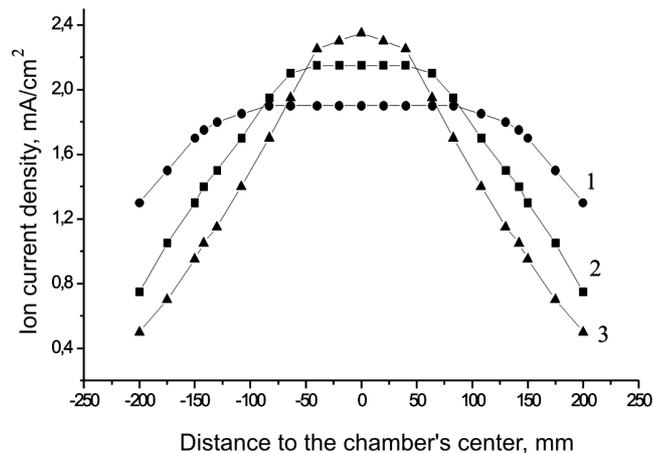


Fig. 3. Radial distributions of the ion current density onto a flat probe located in the vacuum chamber for various pressures in the vacuum chamber: 1 – 0.2, 2 – 0.4, and 3 – 1.1 Pa

diaphragm transmission capacity (Fig. 1). The discharge input power amounted to 1300 W. As the pressure grew, a gradual tendency for the arc discharge to be constricted along the chamber axis was observed.

Specimens to be subjected to the TCT were arranged on a small, electrically insulated stage located at the chamber's center. The stage temperature was measured by a chromel-alumel thermocouple with a corresponding high-voltage decoupling.

By its parameters, argon-nitrogen plasma generated by the discharge allowed the ionic current density on the specimens' surface to be maintained within the range of 1–5 mA/cm². A positive bias voltage of about 150 V and a negative one down to –1200 V can be applied to the specimens, as well as to the whole stage. This allowed us, by periodically changing the bias polarity during the whole process of nitriding, to create the optimum conditions for the initial electronic heating up of a specimen and for maintaining its temperature at an optimum level of 540 °C, as well as for the ionic cleaning of the surface in the course of nitrided layer formation.

The efficiency of the nitriding of disk-shaped specimens 20 mm in diameter and 5 mm in thickness fabricated of constructional steels 40Kh and St45, tool steels R6M5 and R18, and martensitic steel 40Kh13 was studied. Before nitriding, the specimen surface was activated, which included a destruction of oxide films and ionic cleaning, and heated to a working temperature of 540 °C by bombarding the surface with plasma ions for 20 min, provided that a negative bias voltage of about 1000 V was applied.

The procedure of surface nitriding in the arc discharge was carried out in both pure nitrogen and argon-nitrogen mixtures with various argon-to-nitrogen content ratios (30/70, 50/50, 70/30, and 90/10 wt.%); the pressure in the vacuum chamber was 0.1–1.5 Pa, the discharge current 16 A, the discharge voltage 80 V, and the bias voltage 800 V. The pressure level was controlled by needle leak valves and a controllable diaphragm. The duration of the nitriding process in pure nitrogen plasma was 2 h, and that in argon-nitrogen plasma was 30 min irrespective of the plasma content. After the termination of the process, the specimens were cooled down to room temperature in the working chamber in vacuum.

The structure and the phase composition of the diffusion layer was controlled from experiment to experiment on the basis of a variation of the key working parameters of the nitriding process; these included the pressure and the composition of the saturating environment, the specimen temperature, the duration of the process, and the electric characteristics of the discharge.

2.2. Glowing discharge

To make a comparison of the efficiencies of surface modification processes, the experimental researches, where similar specimens were also nitrided but in a dc glowing discharge, have been carried out as well. As in the case with arc discharge, the chamber depicted in Fig. 1 was used. Again, the saturating medium was either pure nitrogen or its mixture with argon in the same ratios. However, the range of working pressure in the chamber was considerably higher: from 13 to 250 Pa. Experiments were carried out in a forevacuum pumping mode: the chamber was first pumped out to a pressure of 0.2 Pa, then the working gases were let into it. The earthed body of the working chamber served as an anode of glowing discharge; the specimen stage supplied by a voltage from –200 to –1200 V was a cathode. Heating of the specimens to the working temperature and their surface cleaning were carried out in the argon environment at a pressure of 40 Pa and a voltage of –1100 V. In the course of experiments, the pressure in the working chamber was maintained at a level of 150 Pa, as a rule. The duration of nitriding was 30 min.

The current-voltage characteristics of a glowing discharge at various pressures of working gases in the chamber are exhibited in Fig. 4. Here, identical currents are expectedly reached at much higher voltages, than it was in the arc-discharge case (Fig. 2).

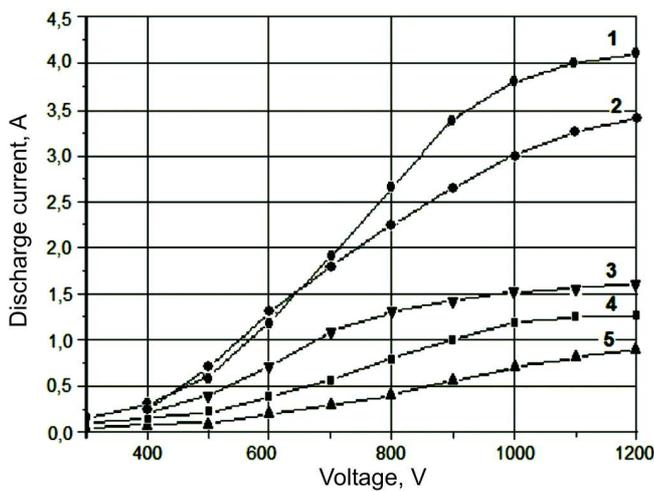


Fig. 4. Current-voltage characteristic of a glowing discharge: in argon at pressures of 50 (1) and 13 Pa (4); in nitrogen at pressures of 100 (2), 50 (3), and 13 Pa (5)

3. Main Results of Experimental Researches

In the course of experiments, the structural features, the phase composition, and the microhardness of specimens nitrided in argon-nitrogen plasmas of a volume arch discharge and a dc glowing discharge were studied.

To study the microstructure of surface layers, the method of optical metallography was engaged, as well as the method of X-ray diffraction analysis of structural-phase transformations in steels modified by nitrogen; in the latter case, a DRON-2 diffractometer was used. The intensity profiles were registered for the monochromatic Fe K_{α} -radiation emission, at a voltage of 30 kV, and at an anode current of 20 mA. X-ray recording was carried out with the Bragg-Brentano focusing in the scanning mode.

The microhardness of specimens was determined on a PMT-3 device at a loading of 0.98 N (HV 0.1). Transverse microsections for metallographic researches were produced following standard techniques. While studying the properties of a nitrided layer, its total thickness (a distance from the specimen's surface to a depth where the hardness was equal to the initial one) was estimated.

The dependences of the microhardness of a nitrided layer on the composition of gas mixture (argon/nitrogen) for steels 40Kh13, 40Kh, St45, and R18 are exposed in Fig. 5. As follows from the results obtained, the microhardness curves demonstrate an extremum at an argon/nitrogen ratio of 70/30 wt.%. The microhardness of steel-R18 specimen surface became twice as large in

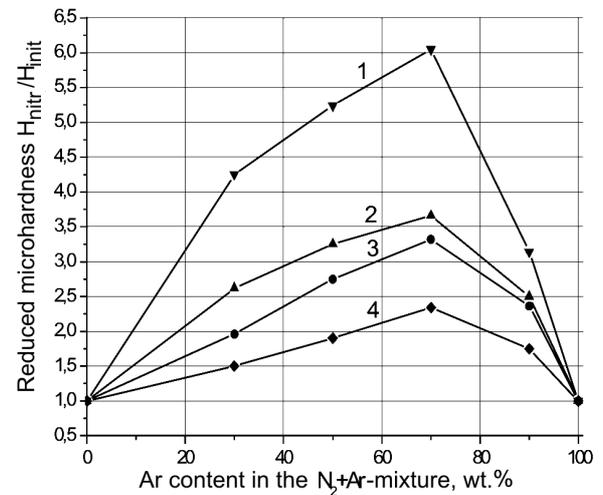


Fig. 5. Dependences of the microhardness of a nitrided layer on the argon-nitrogen gas mixture content: 1 – 40Kh13, 2 – 40Kh, 3 – steel 45, and 4 – R18

its maximum, while that of the steel-40Kh13 surface by a factor of 6. Nitriding the specimens in pure nitrogen and providing the same treatment parameters did not result in an appreciable change of the initial microhardness of the specimens. In this case, only a prolongation of the process to 120 min increased the microhardness of steel-R18 specimens by a factor of 1.4, and that of steel-40Kh13 specimens by a factor of 3.5. This circumstance evidences a lower saturating capability of the technological atmosphere consisting of pure nitrogen.

By changing the key technological parameters of the nitriding process (temperature, duration, pressure, and the composition of a gas mixture), one can obtain a nitrided layer with the required thickness, phase composition, structure, and hardness. In particular, the X-ray diffraction analysis of the modified layer in specimens treated in the pure nitrogen environment revealed a nitride zone on their surface which consists of a high-nitrogen ϵ -phase; the latter interferes with the penetration of atomic nitrogen into deeper layers. The diffusion coefficient of atomic nitrogen in the ϵ -phase, D_{ϵ} , is known to be 60 times lower than that in the α -phase; therefore, an increase of the ϵ -phase layer thickness brings about a reduction of a diffusion flow into the nitrided layer [3]. Hence, the results obtained testify that the nitriding of specimens in the pure nitrogen atmosphere does not provide a maximal possible effect.

Generally speaking, the nitriding of every steel grade has its specific features which are predetermined, in particular, by a capability of nitrogen to form various compounds not only with iron, but also with doping

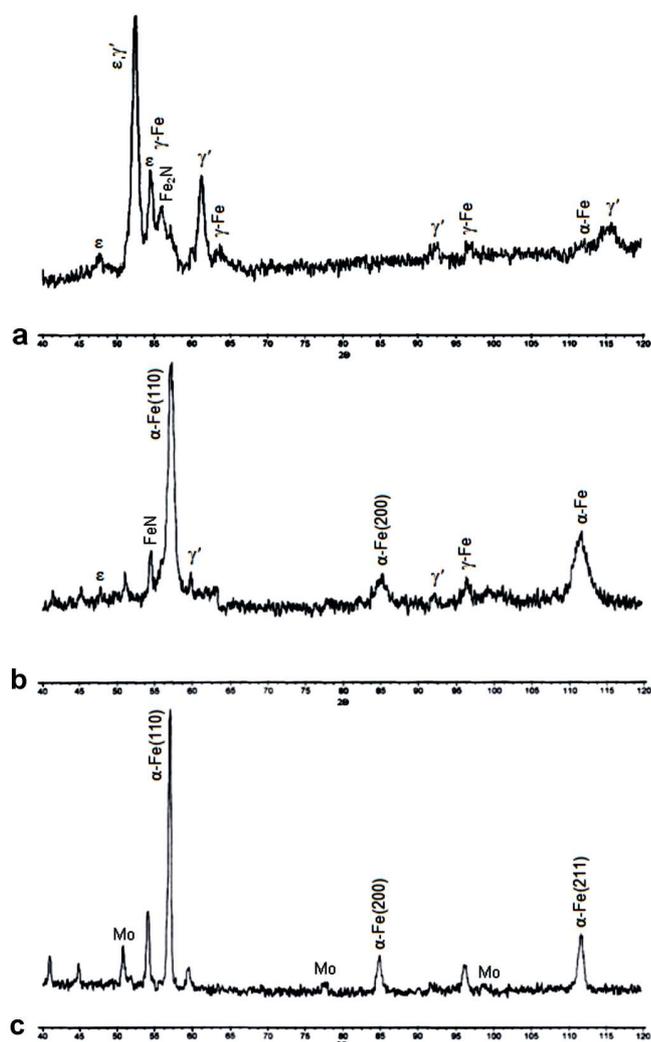


Fig. 6. Diffraction patterns of a surface layer in steel R6M5: *a* – nitrided in a volume arc discharge, *b* – nitrided in a glowing discharge, and *c* – in the initial state (before nitriding)

components which, in their turn, can be in various phase states in the structure of the metal surface layer. For instance, according to the data of X-ray diffraction analysis, the surface layers of fast-cutting R6M5 steel specimens, which were modified in a pure nitrogen environment, contain an ϵ -phase of the $(\text{Fe,Me})_2\text{N}$ type, nitrides of doping elements, carbides (or carbonitrides) of the $\text{Fe}_3(\text{W,Mo})_3(\text{C,N})$ type (in R6M5 steel, these are present as redundant phases), and an α -phase; the latter is a solid solution of carbon and nitrogen in α -Fe (martensite). As nitrogen dissolves in argon to 40–60 at.% of N_2 , the formation of the ϵ -phase becomes blocked.

As an example, in Fig. 6, X-ray diffraction patterns of the surface layers of R6M5 steel specimens nitrided in the plasmas of a volume arc discharge and a glowing discharge, as well as in the initial state, are shown. The main difference between those diffraction patterns is the emergence of the zone of a nitrogen solid solution in martensite, if the nitriding is carried out in the volume arc discharge.

In general, the results of X-ray diffraction analysis allow a conclusion to be drawn that it is possible to control the phase composition of the layer which is formed in the course of nitriding, by introducing an inert gas into the composition of a nitrogen-containing environment, and to obtain the desired structure according to the specific conditions of the operation of details. For example, a nitrided layer on the basis of a nitrogen solid solution in martensite and without the formation of an external nitride zone (it is an optimum for the tool exploitation under the conditions of periodic impact loads) can be obtained by nitriding the tools in the environment of 30 at.% N_2 + 70 at.% Ar. At the same time, a structure optimal for continuous cutting can be obtained by nitriding the tools in the environment of 60 at.% N_2 + 40 at.% Ar.

It should be emphasized that the data of X-ray diffraction analysis are in good agreement with the results of metallographic researches. For instance, the microstructure of the surface layer in specimens nitrided in a pure nitrogen environment or in a gas mixture of argon with a high (up to 90%) nitrogen content pronouncedly manifests a continuous nitride layer 0.5 to 1.5 μm in thickness.

The main difference between the layers modified in a glowing and a volume arc discharge is a more developed nitride zone with high microhardness in the case of a glowing discharge and more plastic layers with a larger diffusion zone – and, sometimes, with lower microhardness – in the case of a volume arc discharge.

4. Discussion of Research Results

Hitherto, there is no common viewpoint concerning the mechanism of diffusion saturation with nitrogen. Most of the authors assert that the crucial role in nitriding belongs to the ions of molecular and atomic nitrogen [4–6]. Special researches [7] demonstrated that the higher concentration of atomic nitrogen is mainly connected with the increase of the discharge voltage to 1000 V and above (however, this method is not always allowable because of a considerable sputtering from treated surfaces). It was also found that the admixing

of argon to the content of the saturating environment exerts a substantial influence on the phase composition and the nitrogen concentration in the nitrated surface layer [8, 9]. In particular, an enhanced concentration of atomic nitrogen is also able to affect the process of nitrating in the mixtures of nitrogen with inert gases in gas-discharge plasma. Such a concentration can be elaborated in the course of ion-molecular reactions with the participation of nitrogen molecules – for instance, $\text{He}^+ + \text{N}_2 \rightarrow \text{N}^+ + \text{N}^0 + \text{He}$, $\text{Ar}^+ + \text{N}_2 \rightarrow \text{N}_2^+ + \text{Ar}$ or $\text{Ar}^+ + \text{N}_2 \rightarrow \text{N}^+ + \text{N}^0 + \text{Ar}$ – and due to the electron impact dissociation. Truly speaking, a practically arbitrary inert gas can be used to mix with nitrogen, though argon is most suitable from economical reasons. It should be noted that the interaction of particles in plasma with the participation of molecular gases is characterized by a multiplicity of specific reactions [10–12].

Now, the substantial attention of scientists throughout the world is attracted to studying the ion-molecular reactions which run in the course of etching and the processes of material surface modification. The materials of the last International Conference on Phenomena in Ionized Gases convincingly testify to that. In particular, it was pointed out that the efficiency of such processes in nitrogen plasma is mostly governed by the particles composed of atomic nitrogen in a metastable state [13] (see also work [14]). The metastable state of an atom or a molecule is such a state, which cannot decay with the emission of a photon, or it has a significant characteristic time of radiation emission. Usually, this is the lowest excited state of an atom or a molecule, the dipole radiation transition from which into an even lower or the ground state is forbidden. Nitrogen atoms have two characteristic radiation lifetimes of 1.4×10^5 and 6.1×10^4 s in their two metastable states [15]; they are many orders of magnitude longer than the lifetimes for ordinary radiation transitions, for which the corresponding values amount to $10^{-7} - 10^{-8}$ s. Therefore, atoms in their metastable state are one of the best agents to transfer the excitation energy obtained by a particle in plasma to the surface of a solid. Really, a typical free path of neutral particles in a nitrogen atmosphere with the pressure ranging from 0.1 to 150 Pa changes from 36 to 2.3×10^{-2} cm, respectively (provided that the diameter of nitrogen molecule is equal to 3.5×10^{-8} cm [16]). Hence, an atom in its metastable state has plenty of chances to get from the plasma region (the theoretically calculated character of the plasma distribution near a solid surface is illustrated by Fig. 2 in work [17]) to the surface of a solid without collisions. It is collisions that are capable

to transit an atom from a metastable into the ground state.

The diagnostics of processes running in plasma in the region of its interaction with the surface of a solid and the determination of the concentration of neutral and, the more so, excited particles are the rather complicated tasks for a plasma experiment (see work [14]) and can comprise the next step of our researches.

However, irrespective of all that, a few facts following from this study should be emphasized. Our researches demonstrated that the main advantage of the nitrating of solids in the plasma of a volume arc discharge is a possibility to form nitrated layers with a considerable thickness, which cannot be obtained within a comparable time interval by any other different way, including nitrating in a glowing discharge. This circumstance testifies that the most optimum conditions for the intensive diffusion of nitrogen are created at the nitrating in the plasma of a volume arc discharge, which, in our opinion, is associated with a high concentration of atomic nitrogen in the near-surface layer of the saturating environment, and, especially, provided that an admixture of inert gas is present.

Effective surface strengthening of alloys by nitrogen under the gas discharge conditions can be achieved only if the initial gas environment with the maximal saturating ability is created. For this purpose, hydrogen and oxygen must be completely excluded from the working environment. For example, the concentration of N^+ ions, which play the dominant role in ionic nitrating of metals, becomes half as large (down to 11%) in the presence of 1 vol.% of oxygen, so that iron is not nitrated at all [18]. Adding even 2 vol.% of hydrogen to nitrogen also results in a two-fold reduction of the concentration of dominant N^+ ions (from 57.6 down to 29%) [19]. We emphasize that the technologies proposed here, unlike the traditional ones – e.g., those which make use of ammonia, – completely satisfy a tendency of changing over to hydrogen-free environments. Owing to the exclusion of a furnace equipment from technological processes, the vacuum technologies are energetically effective and ecologically clean (see also work [12]). The ion-plasma nitrating technology possesses a number of advantages not only from the viewpoint of power consumption, but also with respect to the “know-how” issues. In contrast to the majority of other methods, it belongs to the group of final treatment processes, not demanding therefore such final treatment operations as the grinding and polishing of details. The advantages of the nitrating-in-vacuum method manifest themselves in a substantial

reduction of the consumption of a working gas (by a factor of 20 to 100) and the electric power (by a factor of 1.5 to 4). The rate of treatment becomes 3 to 5 times higher in comparison with the traditional TCT methods. For some details with a complicated shape, the nitriding in vacuum is a unique way to obtain a diffusion layer on a finished product, with a total absence of temperature-induced deformations emerging due to TCT.

However, a few shortcomings are also inherent to the process of nitriding in a glowing discharge. It has a rather low energy efficiency owing to a high voltage of discharge burning (400 to 1100 V) and runs at relatively high pressures of saturating gases (10 to 1000 Pa). This process is accompanied by the cathode sputtering of detail's surface and its contamination. If there are sharp edges and agnails on the surface, a glowing discharge can transform into an arc one. The cathode spots produced by the latter give rise to surface erosion, which considerably exceeds the harmful effect of cathode sputtering. If there are apertures in details to be treated, the effect of hollow cathode manifests itself, which results in a local overheat of details.

The problem of a reduction of both the working pressure and the discharge burning voltage can be solved taking advantage of a technology based on applying the plasma of a volume arc discharge. This process is characterized by low working voltages of about 50 V and low pressures below 1 Pa. The corresponding heating and the cleaning of treated details are efficient very much. Nitriding in a volume plasma allows the energy and the density of an ion flow onto the treated detail to be varied within wide ranges, which provides an opportunity of implementation of an individual approach to the treatment of products fabricated of different tool materials and with different geometries.

5. Conclusions

The process of nitriding in the plasma of a volume arc discharge allows diffusion layers with predetermined composition and physicomechanical properties to be formed on the solid surface. By varying the amplitude and the polarity of the electric potential applied to nitrided details, the pressure and the content of the working mixture, and the arc discharge current, it is possible to create conditions for the formation of a nitrided layer with an optimum structure and the surface purity practically identical to that in the initial state of the surface. This circumstance also provides a possibility

for the individual approaches in the treatments of products with different geometries.

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ПЛАЗМОВА МОДИФІКАЦІЯ ПОВЕРХНІ
КОНСТРУКЦІЙНИХ МАТЕРІАЛІВ

О.Г. Дідик, В.А. Жовтянський, В.Г. Назаренко, В.О. Хомич

Резюме

Описано методи плазмової модифікації поверхні металів, що застосовуються у машинобудуванні. Виконано аналіз переваг

перспективних іонно-плазмових покриттів. Наведено результати досліджень об'ємного дугового розряду з плазмовим джерелом електронів, виконаним на основі порожнистого катода з газоманетронним розігріванням. Розглянуто переваги та недоліки, а також перспективи розвитку енергоефективних плазмово-дифузійних технологій азотування на основі жевріючого та об'ємного дугового розрядів.