Досліджено вплив зміни технологічних параметрів на зміну властивостей зміцненого шару сталі 38Х2МЮА. Встановлено, що глибина дифузійного шару становить 20–620 мкм, поверхнева твердість – 8–12 ГПа при температурах азотування 500–560 °С і тривалості 1–12 годин. Отримано математичні моделі, номограми глибини шару і поверхневої твердості залежно від змін температури і тривалості іонного азотування

Ключові слова: хіміко-термічна обробка, іонне азотування, глибина дифузійного шару, поверхнева твердість

Исследовано влияние изменения технологических параметров на изменения свойств упрочненного слоя стали 38Х2МЮА. Установлено, что глубина диффузионного слоя составляет 20–620 мкм, поверхностная твердость – 8–12 ГПа при температурах азотирования 500–560 °С и длительности 1–12 часов. Получены математические модели, номограммы глубины слоя и поверхностной твердости в зависимости от изменений температуры и длительности азотирования

Ключевые слова: химико-термическая обработка, ионное азотирование, глубина диффузионного слоя, поверхностная твердость

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MODELING OF THE CASE DEPTH AND SURFACE HARDNESS OF STEEL DURING ION NITRIDING

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1. Introduction

Nitriding is the most common and effective method of surface hardening. Such thermochemical treatment can improve the surface hardness, contact endurance, abrasion and adhesion resistance, as well as heat resistance and corrosion resistance of a wide range of machine parts. This process of surface hardening has found its application in many fields of modern mechanical engineering.

Operational requirements for parts produced a need to replace the high-temperature methods of thermochemical treatment (carburizing, high-temperature nitrocarburizing, etc.) with the processes of hardening at lower temperatures (500–650 °C), namely nitriding. This replacement is due to the latest technological developments in the field of various nitriding methods Modern scientific developments allow to gradually eliminate disadvantages of nitriding such as considerable duration, high fragility of the case, insufficient contact endurance values, complexity of the process [1].

Despite the fairly widespread application of the nitriding process in various fields of mechanical engineering, there are a lot of unresolved issues, mainly related to the structure formation mechanism of the case [2]. The structural features of the nitrided case and the matrix itself determine the performance of machine parts and, consequently, the choice of steel, pre-treatment technologies, and the nitriding technology directly. Therefore, forecasting and modeling of processes of formation of structure and properties in the case during nitriding is an urgent task.

Modeling of the processes occurring in nitriding is the most effective method for the process development. The use of the calculation methods allows to promptly obtain information about the basic properties of the hardened case, the rate of diffusion and structure formation processes, formation of nitride and carbonitride cases in varying temperature-time conditions. Modeling of various nitrogen processes allows the simple and efficient solution of many technological tasks of supervision and management of saturation of cases, forecasting of the outcomes and the possibility of developing innovative treatment regimes. Accounting of the factors of complexity and costliness of conducting a large number of experiments makes mathematical modeling of nitriding processes especially promising method of research.

2. Literature review and problem statement

One of the methods of modern intensification of various nitriding processes is the application of a wide range of electric gas discharges.

Nitriding with the ionized nitrogen in glow-discharge plasma (ion nitriding) is the most popular method [3]. The plasma is capable of accelerating reactions by increasing the energy of nitrogen ions, additionally activating them as a result of cathode sputtering. The conventional ionization process is improved by the development of the methods of plasma nitriding with a frame, which protects, or with an active screen, which enhances the process efficiency and quality. Plasma nitriding meets environmental requirements. A large number of installations are operated for its implementation [4].

Ion nitriding has a number of advantages over conventional gas treatment process:

– fairly high rate of saturation processes (by 1.5–2 times);

– possibility of controlled nitriding processes while optimizing the phase-elemental composition of the hardened cases and structural features in compliance with the operating conditions of specific parts;

– fairly minor deformation of products during treatment with a high class of surface finish;

– significant efficiency of the process;

– reduced utilization of saturating gases [5].

Nitriding of the 38Cr2MoAl steel in a glow discharge in the nitrogen atmosphere [6], either with or without the emergence of the hollow cathode effect has not led to the surface hardening due to the presence of oxygen in a gas-discharge chamber, which blocks the nitriding process. Introduction of acetylene in the nitrogen-argon mixture contributes to efficient surface saturation of 38Cr2MoAl steel with nitrogen and allows achieving the surface hardness of 1000 HV50 during nitriding (nitrocarburizing) in a glow discharge. Moreover, the thickness of the carbon nitride zone, resulting in a glow discharge with the hollow cathode, is 2–3 times higher.

Plasma nitriding is widely used for improving the properties of the tool surface and wear-resistant surfaces of various products in mechanical engineering.

The technology, developed by the PlaTeg company using unipolar or bipolar pulsed DC plasma, which improves wear resistance and corrosion resistance of the surface while increasing the fatigue strength of steels is outlined [7]. The developed technology has high performance indicators and allows carrying out the processes of nitriding, nitrocarburizing, oxidation, chemical vapor deposition, ultra-fine surface finishing. Another focus of this company [8] is creating low-pressure plasma equipment for surface modification of products by medium- and high-frequency plasma and micro-frequency plasma.

Ion thermochemical treatment has many advantages over conventional methods, but it is very costly. The ELTRO company (Germany) [9] managed to reduce costs considerably by developing the module for ion nitriding with two alternately operating anode worktables, onto which the parts are loaded. Most of the time the module is operating automatically. The machining cycle includes laser engraving and stacking of parts. These operations take no more than 30 seconds for a part.

The authors [10] studied the effect of oxygen on the course of plasma nitriding of chromium alloy steels. Due to air addition, the oxidizing potential of the working gas varies over a wide range. By analogy with the oxidation during nitriding in a gaseous atmosphere, the same processes in a plasma atmosphere in low-chromium steels lead to accelerated growth of the binder. For high-chromium steels, air addition significantly reduces the nitriding efficiency. For these steels, the negative impact of uncontrolled oxygen impurities can be compensated by a sufficiently high portion of hydrogen in the working gas.

Combination of nitriding and aging of Cr-Ni-Mo-Al at 500 °C for 2–8 hours [11] increases the surface hardness up to 1000 HV, forming the nitrided case depth of 100–200 μm. The surface hardness of solid-solution-treated samples after the process under study increases from 30 to 39 HRC, which suggests the possibility of combining nitriding and aging processes. After a given treatment, the samples show no overageing effect. The compound case, formed in plasma nitriding improves the corrosion resistance of steel.

3. Research goal and objectives

The goal of the paper is to study the dependence of the nitrided case depth and surface hardness on the temperature and duration of thermochemical treatment of steel during ion nitriding.

To achieve this goal, it is necessary to solve the following tasks:

– to build and implement the experimental design, providing the possibility of estimating the coefficients of mathematical models in the form of a quadratic polynomial;

– to obtain a graph-analytical description of variations in the case depth and surface hardness, suitable for a process engineer of thermochemical treatment.

4. Materials and methods of experiments

The material for the study was structural heat-resistant alloy steel 38Cr2MoAl (GOST 4543–71). Ion-plasma nitriding of steel samples was carried out in the dissociated ammonia atmosphere.

For the process of ion-plasma nitriding, the NPI 6.6/6 furnace was used. A general view of the saturating installation is shown in Fig. 1 [12].

Fig. 1. A general view of the NPI 6.6/6 furnace for ion-plasma nitriding

Prior to ion-plasma nitriding, samples were washed with gasoline, acetone, dried and placed in the installation. Then, vacuum with a pressure of at least 5 Pa was created in the chamber. The chamber was purged 2–3 times with dissociated ammonia during 5–6 minutes up to a pressure of 200–

600 Pa, and the gas was then evacuated from the chamber up to a pressure of 5 Pa.

The parts were finished by micro arcs, the chamber was filled with ammonia up to a pressure of 50 Pa and a temperature of 390±10 °C was set. Gas pressure was then increased to 1000 Pa and nitriding was carried out at temperatures of 530±30 °C for 1–12 hours. Steel samples were cooled in two stages:

1) cooling to 300–250 °C in the glow discharge (pressure of up to 0.5–1.0 mm Hg);

2) cooling to 150–100 °C under disabled glow discharge. Further, the parts were cooled in air.

The nitrided case depth was investigated by optical microscopy on the MIM-8 microscope by a standard method at different magnifications.

The microhardness variation range from surface values to the hardness values by 50 units higher than the core hardness was taken as the thickness of the hardened case.

The microhardness of the samples was measured at the PMT-3 device at a load of 50, 100 g and exposure of 7–15 seconds by the standard method (GOST 9450–76).

Experiments were carried out at the points of the second-order central orthogonal composite design: ion-plasma nitriding of 38KH2MYUA steel samples was carried out at temperatures of 500–560 °C for 1–12 hours. The average values of the data obtained are shown in Tables 1, 2.

Table 1

The experimental values of the nitrided case depth

Ion nitriding duration, h	Nitriding temperature, °C		
	500	530	560
1	20	25	35
6,5	180	290	350
12	550	590	620

Table 2

The experimental values of the surface hardness of 38Cr2MoAl steel after ion nitriding

Ion nitriding duration, h	Nitriding temperature, °C		
	500	530	560
1	8	8,2	8,5
6,5	12	10	9
12	12	11	9,5

The nitriding temperature (x_1) and thermochemical treatment duration (x_2) were selected as input variables. As output variables – the nitrided case depth (y_1) and surface hardness (y_2) of treated samples of 38Cr2MoAl steel. 10 parallel experiments were conducted at points beyond the design, based on which the average values of y_1 and y_2 were obtained.

5. The results of experiments to determine the nitrided case depth and surface hardness after the ion nitriding

Mathematical models describing the dependence of the nitrided case depth and surface hardness on the temperature and duration of thermochemical treatment of steel during nitriding can be represented as a quadratic polynomial [13]:

$$y_i = b_0 + a_1x_1 + a_2x_2 + \\ + a_3\left(x_1^2 - \beta\right) + a_4\left(x_2^2 - \beta\right) + a_5x_1x_2, \quad (1)$$

where a_i are the estimated coefficients, β is the parameter calculated based on the number of the core points of the composite design 2^{n-p}, the shoulder of the «star» points α and the number of points of the design according to the formula:

$$\beta = \frac{\sum_{j=1}^{N}\left(x_i^j\right)^2}{N} = \frac{2^{n-p} + \alpha}{N}. \quad (2)$$

The procedure for estimating the coefficients of the model, verification and statistical analysis of accuracy are given in the paper [13]. The resulting model of the nitrided case depth, depending on the normalized values of the temperature and duration of thermochemical treatment is as follows:

$$y = 295{,}55556 + 42{,}5085 \cdot x_1 + 280{,}056 \cdot x_2 - \\ -9{,}211 \cdot x_1^2 + 33{,}289 \cdot x_2^2 + 13{,}75 \cdot x_1 \cdot x_2. \quad (3)$$

Significance evaluation of the coefficients of the model using the Student's t-test showed (Table 3) that the coefficients a_3, a_4, a_5 can be considered insignificant.

Table 3

The results of calculation of deviation values $t_{\text{кр}}s_i$ for significance evaluation of the model coefficients

Deviation values $t_{\text{кр}}s_i$		
For linear coefficients	For quadratic coefficients	For pair-interaction coefficients
20,55415	35,59727	25,17107

Under this circumstance, the model of the saturated case depth, depending on the normalized values of the temperature and duration of thermochemical treatment takes the form

$$y = 295{,}55556 + 42{,}5085 \cdot x_1 + 280{,}056 \cdot x_2. \quad (4)$$

The mathematical model describing the effect of the temperature and duration of nitriding on the values of surface hardness of the nitrided case is generally represented as follows:

$$y = 9{,}8 - 0{,}8335 \cdot x_1 + 1{,}30026 \cdot x_2 + 0{,}09853 \cdot x_1^2 - \\ -0{,}80147 \cdot x_2^2 - 0{,}75 \cdot x_1 \cdot x_2. \quad (5)$$

The significance test of coefficients showed that the coefficient a_3 may be deemed insignificant, so the model is converted into

$$y = 9{,}8 - 0{,}8335 \cdot x_1 + 1{,}30026 \cdot x_2 - \\ -0{,}80147 \cdot x_2^2 - 0{,}75 \cdot x_1 \cdot x_2. \quad (6)$$

The response surfaces in factor space in a given design region are shown in Fig. 2, 3.

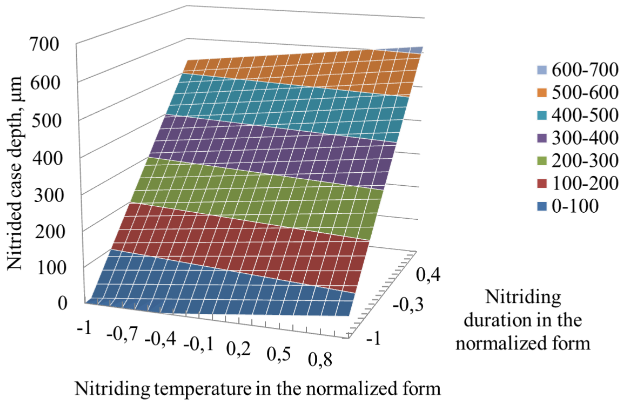

Fig. 2. The response surface in the factor space of variations in the case depth under variations in the treatment temperature and duration

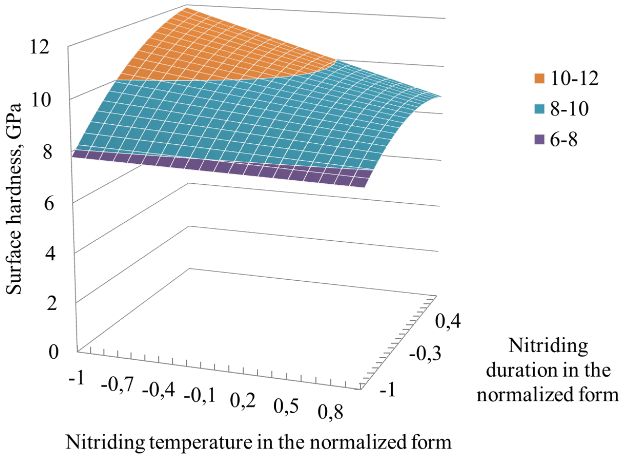

Fig. 3. The response surface in the factor space of variations in the surface hardness after nitriding depending on the treatment temperature and duration

6. Discussion of the results of modeling the effect of temperature and duration of thermochemical treatment on the case depth and surface hardness

As follows from the results obtained (Fig. 2), the optimum values of the nitrided case depth correspond to the region of maximum values of temperature and duration of the process in a predetermined range. For surface hardness, there is another relationship – the region of maximum values corresponds to the maximum duration at the minimum values of treatment temperature in a predetermined range of values (Fig. 3). In both cases, the optimum values mean those on the boundary of the experimental design region.

The justification for such a relationship is in the plane of formation features of nitrides, carbides and carbonitrides of alloying elements and the alloy base – iron, their coagulation and the course of diffusion processes from the sample surface deep into the alloy. At 500 °C, highly hard nitride case is formed for a maximum time on the steel surface, corresponding to the nitride phase. The continuous close-packed nitride case with a high hardness, namely 12 GPa, is a barrier for diffusion processes, which contributes to the termination of the case growth at a given temperature. Increasing temperature of ion nitriding promotes intensification of diffusion processes into the metal, which increases the case depth while reducing surface hardness (up to 9–9,5 GPa).

Based on these data, graph-analytical description of the relationship between input and output variables – nomograms – convenient for a process engineer of thermochemical treatment can be performed. This requires finding the response surface cross-section at a given level of output variables and project it onto the plane of the input variables. Such a description allows estimating the probable nitrided case depth and surface hardness under the joint influence of nitriding temperature and thermochemical treatment duration (Fig. 4, 5).

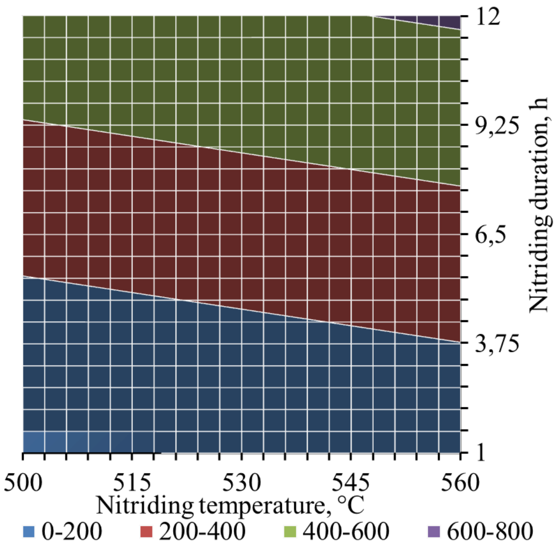

Fig. 4. The nomogram of the case depth under the joint influence of nitriding temperature and duration

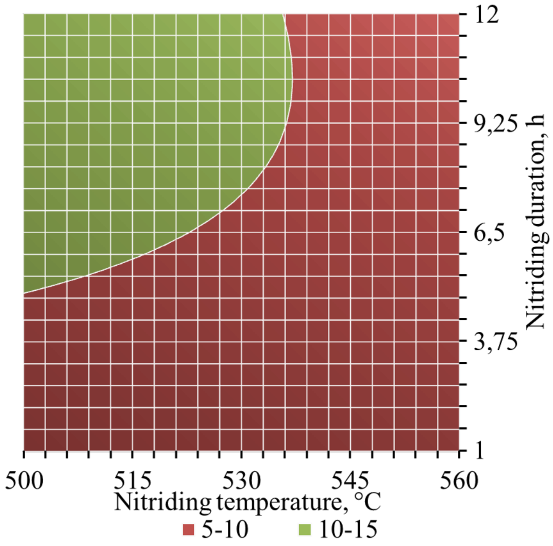

Fig. 5. The nomogram of the surface hardness of 38Cr2MoAl steel under the joint influence of nitriding temperature and duration

Such a description allow identifying the specific conditions of nitriding (temperature and duration), based on the desired nitrided case depth or surface hardness of 38Cr2MoAl steel respectively.

7. Conclusions

1. The case depth varies from 20 to 620 μm in the range of nitriding temperatures of 500–560 °C and duration of 1–12 hours. The surface hardness varies in the range of 8–12 GPa. The mathematical description of the dependence of the nitrided case depth on the temperature and duration of thermochemical treatment of 38Cr2MoAl steel has the form of a linear polynomial. The dependence of the surface hardness of the temperature and duration of thermochemical treatment includes, in addition to linear, the effect of the joint influence of the input variables and the quadratic effect of the thermochemical treatment duration.

2. The graph-analytical description, obtained by the response surface cross-section by the planes of equal values of the output variables, can be a convenient tool for a process engineer of thermal treatment. This kind of description allows estimating the probable nitrided case depth case and surface hardness depending on variations in temperature and duration of the thermochemical treatment of 38Cr2MoAl steel in operational mode.

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