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Optimization of the ion-plasma nitriding of structural steel

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Abstract. Mathematical planning was used in order to determine the optimal mode for ion nitriding the structural steel 0.38C–2Cr–2Ni–Mo. This was justified by the requirement to preserve strictly limited functional parameters obtained as a result of diffusion saturation: hardness in the range of 450–650 HV in a layer 0.15–0.40 mm deep. According to of X-ray diffraction analysis in addition to reflections from the matrix phase (alloyed ferrite α-Fe), the reflexes of nitride compounds are recorded, namely, the γ′-phase with a FCC lattice (Fe_4N) and the ε-phase with a HCP lattice (Fe_3N) with a high nitrogen content. An analytical expression in the form of a linear function for the studied optimization parameters (microhardness and depth of nitration) was obtained by implementing factor planning and regression processing of the obtained data. This made it possible to assess the degree and direction of influence of the investigated factors on the optimization parameters under study.

1. Introduction

As is well known, ion-plasma nitriding is an effective form of diffusive nitrogen saturation for the surface layers of processed products [1–3]. It is carried out in a glow discharge excited on a cathode's surface in a nitrogen-containing gaseous medium (ammonia, nitrogen, etc.) under conditions of deep rarefaction. The saturation process is conducted in a steel container, the anode. Active gas ionization occurs due to the high voltage generated. Nitrogen ions enter the high-tension zone, accelerate to high speeds, and, colliding with the cathode, are embedded in its surface. The high kinetic energy possessed by the nitrogen ions is converted into thermal energy. As a result, the cathode is heated to a temperature of 450–600 °C in a short period of time (approximately 15–30 minutes), at which point diffusion occurs.

Unlike traditional gas nitriding in furnaces, this chemical-thermal treatment reduces the process' overall duration (by 1.5–2 times), makes it possible to regulate the process in order to obtain a nitrided layer with the desired properties, and is more environmentally friendly [4–5].

In particular, this technology is used for nitriding the structural steel 0.38C–2Cr–2Ni–Mo. Formally, this steel is not a classic example of an alloy steel intended for nitriding because it does not contain important elements like aluminum. At the same time, the processing modes currently used are unable to provide the desired combination of functional parameters. The diffusion layer has a hardness value higher than the regulated one and is characterized by insufficient depth. Therefore, in this investigation, it was necessary to find a mode of chemical-heat treatment that provides from this structural steel a hardness in the regulated range of 450–650 HV. It also needs to maintain a layer depth of 0.15–0.40 mm.

Thus, the main purpose of this research is to study the peculiarities of ion-plasma nitriding of 0.38C–2Cr–2Ni–Mo steel and determine the optimal regime for ion nitriding while taking into account whether the specified properties are obtained.

2. Experiment

The object of research is a standard structural steel 0.38C–2Cr–2Ni–Mo, the chemical composition of which is shown in Table 1.

Table 1. Chemical composition of 0.38C-2Cr-2Ni-Mo steel (mass. %).

C	Si	Mn	Ni	Cr	Mo	S	P	Cu
0.36	0.28	0.38	1.46	1.49	0.22	0.008	0.012	0.17

The research samples for ion-plasma nitriding were rectangular parallelepipeds with sides of 10 mm×10 mm×50 mm. In order to obtain a stable microstructure, the samples were annealed at a temperature of 840 °C for 4 hours. The standard heat treatment before nitriding was quenching and high-temperature tempering. Specifically for the studied steel, the quenching temperature was 880 °C with cooling in oil; the tempering temperature was in the range of 560–600 °C.

Chemical heat treatment was performed on an ion-plasma nitriding unit with a YON-50 glow discharge. The hardness of the nitrided surface layer was measured using a DuraScan automatic universal hardness tester that operates in micro-and macro-ranges and has a load of 100 MPa. The nitration's depth was determined on a transverse metallographic sample after etching with a 10 % solution of nitric acid using a metallographic complex, including a GX51 optical microscope and a software unit. A 'D2 PHASER' diffractometer in cobalt K_α-radiation was used for X-ray diffraction studies of phase composition. Phase analysis was carried out using the international diffraction data file ICDD PDF-4 (the International Centre for Diffraction Data) [6].

3. Results and discussion

Initially, the effect of the tempering temperature on the hardness and depth of the diffusion layer after ion-plasma nitriding was determined. Standard heat treatment was used for this steel: after quenching from 880 °C, tempering was carried out for two hours at temperatures of 560 and 600 °C. The preliminary ion-plasma nitriding of the hardened and tempered steel was carried out in a regulated regime: at 525 °C for 90 minutes.

The data acquired on the hardness and depth of the nitration are shown in Table 2.

Table 2. The hardness and depth of nitration after heat treatment in a regulated regime.

№	Quenching temperature, °C	Tempering temperature, °C	Hardness after tempering, HV_{10}	Hardness after nitriding, HV_{10}	Depth of nitration, mm
1	880	600	395	750	0.172
2	880	560	425	775	0.171

The metallographic (see box) and diffraction patterns of the nitrided layer are presented in Figure 1.

The microstructure is a characteristic hetero phase image with the presence of nitride particles, which are mainly spherical. X-ray phase analysis allowed us to identify the phase composition of the steel after ion-plasma nitriding. In addition to reflections from the matrix phase (alloyed ferrite α-Fe), the investigated nitrided layer γ′-phase with a FCC lattice (Fe_4N) and the more nitrogen-rich ε-phase with a HCP lattice (Fe_3N) were fixed.

The results of preliminary experiments have shown that it is possible to effectively influence hardness after nitriding by changing the tempering temperature after quenching. For this reason, it is necessary to conduct a more detailed investigation in which the influence of the factors can be

analyzed over a fairly wide range. It seemed appropriate to conduct experiments using mathematical planning [7, 8].

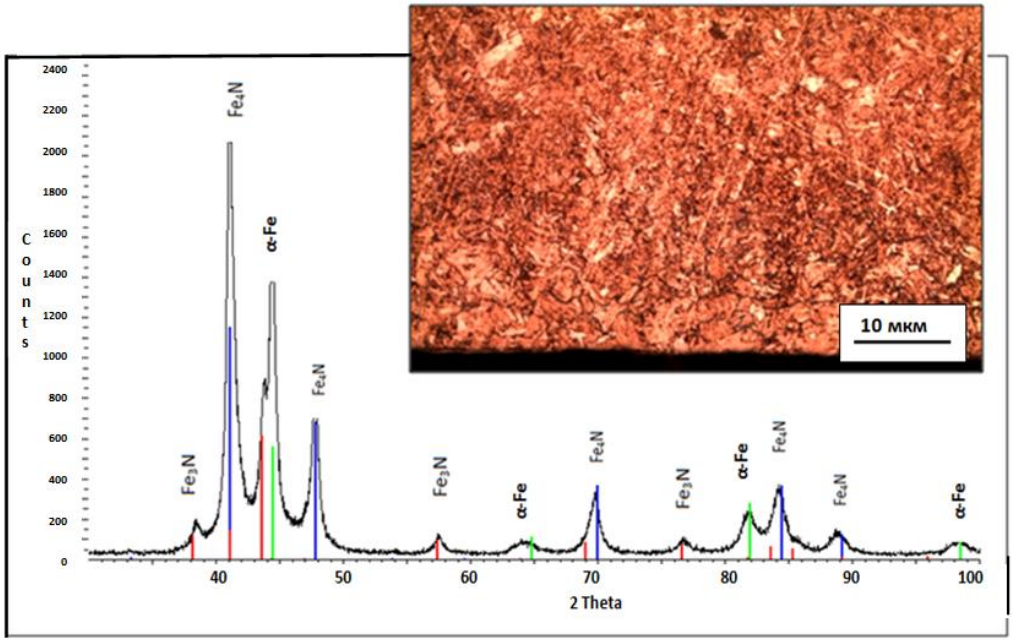

Figure 1. X-ray diffraction and metallographic patterns of 0.38C–2Cr–2Ni–Mo steel after ion-plasma nitriding.

The mathematical planning method is considered effective for solving problems when it is necessary to search for the optimal properties of the research object. The idea behind the method is to conduct a minimum number of experiments while simultaneously varying the values of independent variable factors according to specially formulated rules, to find the optimal region, and to obtain its mathematical expression.

In this article, the following variables were used as the studied factors:

a) the tempering temperature after quenching, °C;

b) the temperature of ion-plasma nitriding, °C;

c) the time for nitriding, min.

Parameters like hardness HV_{10} (y_1) and the depth of the nitration h (y_2) were used as optimization parameters. A complete three-factor type 2^3 experiment was carried out.

For the center of the plan (zero level), the standard parameters of 38Cr2Ni2Mo steel were used:

a) the tempering temperature after quenching, equal to 580 °C (x_1);

b) the nitriding temperature, equal to 525 °C (x_2);

c) the time for nitriding, equal to 60 min (x_3).

Thus, 9 experiments with the experiment in the center of the plan were conducted.

Table 3 shows data on the values of the discussed factors at zero level, the intervals of their variation, and the indicators at the upper and lower levels.

Table 4 provides the planning matrix based on the above factors and variation intervals, as well as the obtained values of the hardness HV_{10} and the depth of the layer h.

Standard statistical processing of the results was performed. After verification of the equation itself and its coefficients for adequacy, a regression dependence was obtained in the form of two linear functions. The final forms of the equations are presented, taking into account the established significance of the regression coefficients. Namely, this is the absence of the effects of paired and triple interaction, as well as the obvious insignificance of the effect of the tempering temperature on nitration depth.

Table 3. Factors and variation intervals.

	Tempering temperature, °C	Factors	Time for nitriding, min
Code	x_1	x_2	x_3
Zero level (0)	580	525	60
Intervals of variation (J)	20	25	30
Upperlevel (+1)	600	550	90
Lower level(–1)	560	500	60

Table. 4. The planning matrix.

Experiment number	x_1	x_2	x_3	Hardness, HV_{10}	Depth of nitration h, mm
1	560	500	30	590	0.072
2	600	500	30	525	0.079
3	560	550	30	640	0.115
4	600	550	30	575	0.139
5	560	500	90	670	0.144
6	600	500	90	605	0.139
7	560	550	90	700	0.191
8	600	550	90	645	0.165
9	580	525	60	650	0.123

Hardness, HV_{10}: $y_1 = 618 - 31x_1 + 21x_2 + 35x_3$; Depth of nitration, h, mm: $y_2 = 0.13 + 89x_2 + 118x_3$

Analysis of the regression equations allows us to evaluate the degree and direction of the influence of the investigated factors on the optimization parameters. The tempering temperature after quenching, as expected, has an effect on the hardness with the opposite sign. However, in general, the impact of all factors is approximately at the same level. The nitriding time has a slightly greater effect on nitration depth. The tempering temperature has no statistical effect, which is reflected in the absence of the x_1 indicator in the equation.

Since the experiment requires a certain combination of nitriding conditions and the required technological parameters (hardness in the range of 450–650 NV and nitration depth in the range of 0.15–0.40 mm), the most acceptable treatment mode is experiment 8 (Table 4).This regime involves tempering at 600 °C (for 2 hours) after quenching, followed by ion-plasma nitriding at 550 °C for 90 minutes. As a result of this treatment, the hardness reaches 645 HV_{10} and the depth of the nitration h-0.165 mm, which satisfies the required performance indicators.

A physical indicator like the diffusion coefficient (D) acquires an important functional value when performing diffusion saturation operations. Therefore, an empirical calculation of this characteristic was performed.

The diffusion coefficient D was calculated using formula [9,10]:

$$D = \frac{x^2}{2\tau}$$

where D – diffusion coefficient; x – average depth of the nitration, cm; τ –the time of diffusion, sec.

Computation was performed for the following experimental conditions:

1. For the center of the plan: the temperature is 525 °C, the saturation time is 60 min, and the depth of the nitration is 0.13 mm. The calculated value of the diffusion coefficient is $D = 2.2 \cdot 10^{-8}$ cm²/ sec.

2. For experiment 8: the temperature is 550 °C, the saturation time is 90 min, and the depth of the nitration is 0.165 mm. $D = 2.5 \cdot 10^{-8}$ cm²/ sec.

The obtained values of the diffusion coefficient are consistent with the available data from the literature describing the coefficient values for iron saturation at different temperatures and phase states [11].

4. Conclusion

The purpose of this work was to study changes in the hardness and depth of the nitration during nitrogen saturation of 0.38C–2Cr–2Ni–Mo steel in an ion-plasma nitriding unit, as well as the selection of optimal nitriding parameters according to specified criteria, namely certain ranges of the diffusion layer's hardness and depth.

To find the optimal regime for nitriding this steel in a glow discharge, experimental research based on mathematical planning was carried out. The hardness values HV_{10} and the depth of the nitration h were used as the studied optimization parameters. At the same time, it was necessary to choose the optimal saturation regime, which provides a regulated range of these characteristics within the limits of HV_{10} 450–650 and h 0.15–0.40 mm. As a result, the required regime of chemical heat treatment was determined. It includes quenching, tempering at 600℃ (for 2 hours), and subsequent ion-plasma nitriding at 550 °C for 90 minutes. The required results were attained: a hardness of 645 HV_{10} and a nitration depth of h 0.165 mm.

Qualitative phase analysis of the samples was carried out via X-ray diffraction. The reflexes observed after ion-plasma nitriding can be interpreted as being obtained by reflection from the matrix phase of α-Fe type FeCr (chromium-doped ferrite). In addition, reflections from the phases are recorded that correspond to nitrate compounds, namely the γ′-phase (Fe_4N) and the more nitrogen-rich ε- phase (Fe_3N).

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