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Investigation of the penetration of the counter-body in the nitride zone of steels and analysis of concomitant characteristics

Angel ZYUMBILEV[1], **Nikolay TONCHEV**[2], **Emil YANKOV**[3,*], **Iliya ZYUMBILEV**[1], **Valentin GAYDAROV**[2]

[1] Technical University of Sofia, Plovdiv Branch, Plovdiv, Bulgaria
[2] University of Transport, Sofia, Bulgaria
[3] University of Ruse "Angel Kanchev", Ruse, Bulgaria
*Corresponding author: eyankov@uni-ruse.bg

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Abstract

The present research summarises the influence of technological modes based on experimental studies of steels BH11 and BH21 (BS 4659) for hot work in ion nitriding. The peculiarity is that one or several precisely defined modes are not fixed, but a group of parameters from the action of the considered technology in a planned experiment is studied. As a result, the technological modes of ion-nitrided steels are determined, corresponding to the minimal and maximal penetration of the counter-body in the nitrided layer. The obtained results are based on additional research related to microhardness and relative wear resistance. In this way a finished idea of the tribological properties of ion nitrided steels with a specialised purpose is obtained. The tools by which the problem is solved are design of experiments (DOE), modelling and single-criteria optimisation of controlled quantities. The main contribution of the paper is a formalised determination of the influence of the technological parameters of ionic nitriding on the phase composition of the nitrided zone and respectively on the wear resistance of the surface layer.

1. Introduction

When applying the materials technological processes related to coatings, layers, or their explored parameters, it is necessary to create an opportunity to control and manage the results of the studied indicators [1]. This circumstance is directly related to the demand for specific applications of the respective technological regimes, characterised by one or another efficiency. The process of managing conditions of change of technological factors makes it possible to determine various useful solutions simultaneously with the group of performed experiments. Another advantage of this idea is that it comprehensively analyses the process or technology in question and thus evaluates the whole set of properties. All this can be achieved through laboratory tests or simulation experiments with varying combinations of modes. As a result, a solution is obtained for the design of optimised processes or systems. These ideas are discussed in detail in [2-4]. In order to optimise the nitriding process, in [5] experimental studies of plasma nitriding of four selected steels are stated. The microstructures were obtained, including the thickness of the nitride zone. The depth profile of austenitic stainless steel after plasma nitriding at a precisely set temperature is analysed in [6]. The different properties of the layer are also proved in [7] where this is confirmed by the different phases of the layer (ε, ε + γ', γ'). The results of the experiments show that the structures of the nitrided layers differ for the different parameters of the mode and this determines the properties of the surface layer. A similar research was done in [8].

The analysis of the bibliography proves that all considered sources, in this case, are based mainly on experimental research, the characteristic of which is that it is carried out under a fixed one or several well-defined modes. With such a statement of the problem, the general optimal value of the studied group of parameters in the operation of the considered technology cannot be proved.

2. Purpose of the research

The present research aims to determine the technological modes of ion nitrided steels BH11 and BH21 (BS 4659) for hot work, corresponding to the minimal and maximal penetration of a counter-body in the nitrided layer. The obtained results are related to our previous studies related to microhardness and relative wear resistance. In this way, a finished idea of the tribological properties of ion nitrided steels with a specialised purpose is obtained. The tools means to solve the problem are design of experiments (DOE), modelling and single-criteria optimisation of controlled quantities.

3. Problem statement

Steels BH11 and BH21 (BS 4659) for hot work were selected for testing. The chemical composition in weight percent of these steels is listed in Table 1.

The steels were quenched in a vacuum furnace and gas-cooled with 6 bar argon, according to Table 2. Thus treated, the samples were ground to *Ra* = 0.32 µm and then nitrided. According to changing combinations of parameters from Table 2 samples of the tested steels were subjected to the design of experiments (DOE) described in [9-11]. DOE is realised through a planned experiment in which pre-planned deviations of the factors from the normal mode of the process are realised, in order to explore their influence on the initial value in a limited number of experiments. An important requirement for the factors is that they should be compatible. The concept of code defined in Table 2 represents the normalisation of the respective parameter in accordance with its bounds. All the following equations are realised in coded form. A complete factor experiment of 2^4 type was conducted for the studied steels, with the following input factors: nitriding temperature (X_1); ammonia pressure in the vacuum chamber (X_2); treatment duration (X_3); temperature of tempering (X_4), and target parameters: the penetration of the counter body (h) into the nitride zone of the nitrided layer, the relative wear resistance of the nitrided layer (Kv) and the microhardness of the layer (HV 0.1).

By the developed methodology for accelerated abrasion resistance testing, the degree of wear of ion nitrided samples from the planned experiment and samples processed under different heat treatment modes was determined [4]. The scheme of the description for the research of the relative wear resistance and the penetrating counter-body is shown in Figure 1a. Diamond coating is applied to the cylindrical surface of the roller, which slides along the plane of the surface of the test sample and the reference standard at continuous coolant pouring. Three tests were performed on each sample piece and reference standard, and the results were averaged.

The test parameters are as follows: size of the samples 20 × 20 × 10 mm; diameter of the counter-body 52 mm; width of the work surface 1 mm; radial beating of the working surface 4 µm; counter-body rotation frequency 570 min^{-1}; peripheral speed 1.5 m/s; test duration 180 s; coolant 0.5 % aqueous solution of K_2Cr2O_4;

Table 1. Chemical composition of steel from the class (wt. %)

Steel	C	Si	Mn	Cr	Mo	W	V	Ni	Cu	S
BH11	0.38	0.91	0.22	4.5	1.20	–	0.47	0.14	0.11	0.006
BH21	0.3	0.18	0.26	2.7	–	8.01	0.29	–	–	0.015

Table 2. Intervals of change of technological parameters

No.	Parameter of the technological mode of ion nitriding and preliminary heat treatment	Extreme low		Average		Extreme upper	
		Interval value of mode change					
		Code	[-1]	Code	[0]	Code	[+1]
1	Nitriding temperature t_{nit}, °C (X_1)	510		530		550	
2	Ammonia pressure P_{NH3}, Pa (X_2)	150		300		450	
3	Time of nitriding τ, h (X_3)	4		7		10	
4	Temperature of tempering t_{tem}, °C (X_4)	600		650		700	

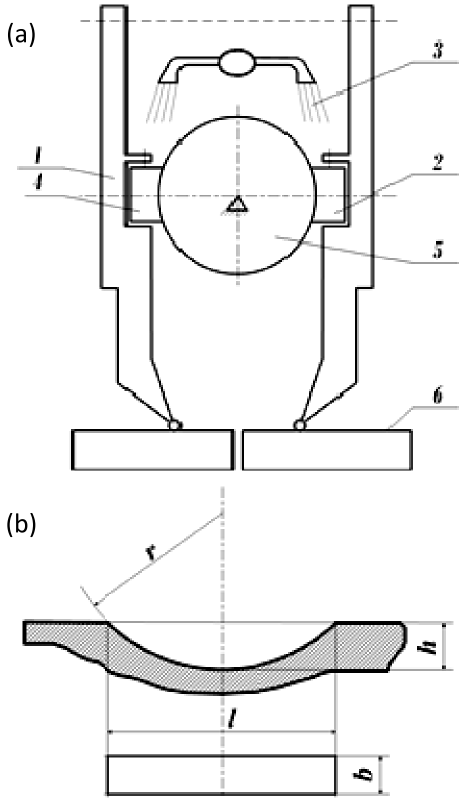

Figure 1. Testing scheme for: (a) sample (1 – load arms; 2 – tested sample; 3 – liquid; 4 – reference standard sample; 5 – counter-body (roller); 6 – holders) and (b) cavity obtained after testing the nitrided wear specimens

coolant temperature 24 ± 1 °C; load 20 N; counter-body with a dispersed coating of nickel-phosphorus bond with a grain size of 3 – 5 µm; reference steel BH10, hardness HRC 51 ± 1. The tests were performed at room temperature.

The evaluation of the test results was performed according to the relative wear resistance:

$$Kv = \frac{V_{\mathrm{sam}}}{V_{\mathrm{ref}}}, \quad (1)$$

defined as the ratio of the worn volumes, the sample material (V_{sam}) and the material of the reference standard (V_{ref}).

After carrying out the tests, following the defined wear pattern, a recess (*h*) was obtained on the surface of the nitrided sample and the reference standard (Fig. 1b). Using an instrumental microscope, its length (*l*) was measured to the nearest 0.010 mm. The following approximate dependence:

$$h = \frac{l^2}{8r} \quad (2)$$

exists between the size of the recess and its chord, and this dependence is used to determine the depth of penetration of the counter body (the roll) into the nitrided layer.

To clarify the morphological features of the nitrided layers, a metallographic analysis was performed. In determining the structure and thickness of the resulting layers, the Axioskop microscope was used, through which the metallographic photographs were taken. The thickness of the nitrided layer was determined by the depth to which microhardness equal to the core plus 50 was achieved. The measurement of the microhardness of the obtained layers was performed with a Leitz microhardness tester, with a load of 0.98 N (100 g), according to the Vickers method. The test sections were developed with a 3 % solution of nitric acid in ethyl alcohol. The X-ray diffraction analysis was performed on an X-ray diffractometer “DR0H-1” in cobalt radiation. Qualitative X-ray diffraction analysis was performed to determine the available phases in the surface-bound zone.

The maximal surface hardness and the total thickness of the nitrided layer were determined by the measured microhardness of vacuum-thermally and ionically nitrided samples in-depth, and the thickness of the nitride zone was determined with the help of metallographic analysis. All experimental results of the investigated quantities are presented in [12].

4. Results of the research and discussions

Depending on the chemical composition of the steels and the nitriding modes, the relative wear resistance, the depth of penetration of the counter-body and the microhardness vary within different limits. The positive effect of ion nitriding on the wear resistance of the studied steels is associated with the presence of alloying elements (V, Mo, Cr, W) in the solid solution and its increased resistance after nitrogen saturation, as well as the slow coagulation of nitride particles. That is why the improved wear resistance of the surface nitrided layer should not be considered separately and outside the context of the complex of operational characteristics of the nitrided parts. In [8] the results for abrasive wear resistance (*Kv* – relative wear resistance of the test bodies, *h* – penetration of the counter-body in the nitride zone of the nitrided layer) and the microhardness of the nitrided steels are given. Based on these results, using the method [9], the following significant adequate regression models for the size of the cavity *h*, which is obtained due to the penetration of the counter-body in the bound zone of the nitrided layer, for microhardness and relative wear resistance are derived.

They are as it follows:

- for steel BH11

$$h = 7.97 - 1.16\,X_1 - 0.325\,X_2 - 0.99\,X_3 - 0.4\,X_4 + 0.34\,X_1X_3 + 0.28\,X_1X_4 - 0.68\,X_2X_3 + 0.21\,X_2X_4 + 0.38\,X_3X_4, \quad (3)$$

$$HV\,0.1 = 11914.4 + 254.13\,X_1 - 143.65\,X_2 + 134.44\,X_3 - 274.03\,X_4 - 51.50\,X_1^2 + 248.75\,X_1X_2 - 88.75\,X_1X_3 - 136.25\,X_1X_4 - 624\,X_2^2 - 301.25\,X_2X_3 + 76.25\,X_2X_4 + 194\,X_3^2 - 36.25\,X_3X_4 - 144\,X_4^2, \quad (4)$$

$$Kv = 0.4020 - 0.0828\,X_1 + 0.0033\,X_2 - 0.0523\,X_3 - 0.0008\,X_4 + 0.0037\,X_1^2 + 0.0106\,X_1X_2 + 0.0244\,X_1X_3 - 0.0006\,X_1X_4 + 0.0087\,X_2^2 - 0.0469\,X_2X_3 + 0.0031\,X_2X_4 + 0.0237\,X_3^2 + 0.0093\,X_3X_4 + 0.0512\,X_4^2. \quad (5)$$

- for steel BH21

$$h = 8.94 - X_1 - 0.26\,X_2 - 0.78\,X_3 - 0.14\,X_1X_2 + 0.33\,X_1X_4 - 0.74\,X_2X_3 + 0.16\,X_2X_4 - 0.47\,X_3X_4, \quad (6)$$

$$HV\,0.1 = 11074.8 - 92.33\,X_2 + 97.99\,X_3 - 514.46\,X_4 + 117\,X_1^2 + 86.25\,X_1X_3 + 311.25\,X_1X_4 - 93\,X_2^2 - 273.75\,X_2X_3 - 173.75\,X_3X_4 - 85.50\,X_4^2, \quad (7)$$

$$Kv = 0.3804 - 0.0804\,X_1 - 0.0076\,X_2 - 0.0504\,X_3 + 0.0300\,X_4 + 0.0156\,X_1^2 + 0.0050\,X_1X_2 - 0.0025\,X_1X_3 + 0.0037\,X_1X_4 + 0.385\,X_2^2 - 0.054\,X_2X_4 - 0.029\,X_3^2 - 0.037\,X_3X_4 + 0.059\,X_4^2. \quad (8)$$

The obtained regression models (3) and (6) for both steels have a high power correlation coefficient (R = 0.85 – 0.91). They are adequate according to Fisher's criterion: $F_{Calc} = 5.16 > F_{Table} = 4.1$ for model (1); $F_{Calc} = 4.8 > F_{Table} = 3.43$ for model (6), at a significance level of 0.05. Due to the models (3) and (6) in Table 3, the modes of minimal and maximal penetration of the counter-body are determined using the methodology described in [13].

From the analysis of models (3) and (6), the negative values before X_1, X_2, X_3 and X_4 show that increasing of the nitriding temperature (X_1), ammonia pressure (X_2) in the chamber, duration of treatment (X_3) and annealing temperature (X_4) reduces the size of the recess in the nitrided layer (h), and increases the relative wear resistance (Kv) of the obtained nitrided layers.

This is explained by the fact that the change of the control parameters (nitriding temperature, ammonia pressure in the chamber and duration of treatment) of the ion nitriding process leads to the formation of nitrided layers with a certain thickness (7 – 11 μm), microhardness (1048 – 1149 HV 0.1) and phase composition (γ', ε + γ') of the nitride zone. It can be noted that steel BH21 after annealing at 600 °C has the smallest size of the recess h = 6.6 μm at the best relative wear resistance Kv = 0.34 after nitriding at: temperature 550 °C, duration of processing 10 h and pressure of ammonia in the chamber 450 Pa.

The resulting nitrided layer has a microhardness of 1097 HV 0.1, which gradually changes in the depth of the layer, and the thickness of the bound zone is 9 μm and has a mixed composition (ε + γ') phase. When testing the test bodies, the counter-body penetrated to a depth of 6.6 μm into the nitride zone, which is less than its thickness. This shows the high wear resistance of the biphasic (ε + γ') nitride zone in which the ε-phase is greater than the γ'-phase. Despite the lower microhardness of the formed bonded (white) zone, its relative wear resistance is the highest. This is due to the presence of the ε-phase, which has a hexagonal lattice and has a lower coefficient of friction than the other phases. A significant role is played by the uniform distribution of the microhardness in the depth of the nitrided layer, obtained in this nitriding regime indicated for the tests of steels in Figure 2.

Table 3. Modes for the penetration of the counter-body into the nitrided layer

Extremum	Steel	Value h, μm	Processing mode			
			t_{nit}, °C	P_{NH3}, Pa	τ, h	t_{tem}, °C
Min	BH11	4.68	550	450	10	600
Max		11.66	510	450	4	600
Min	BH21	6.67	550	450	10	600
Max		11.64	510	450	4	700

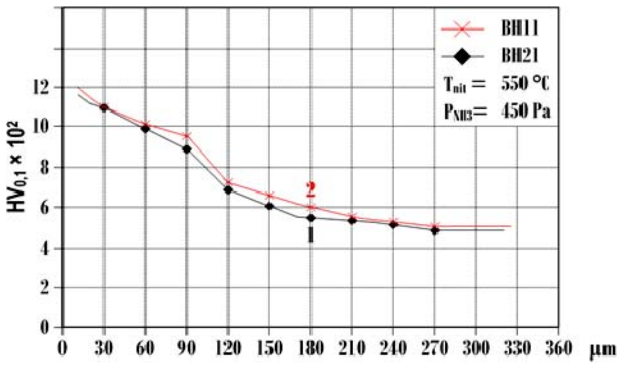

Figure 2. Distribution of the microhardness in the depth of the nitrided layer for the tested steels at: t_{nit} = 550 °C; P_{NH3} = 450 Pa; τ = 10 h

5. Conclusions

Regression models have been derived, reflecting the relationship between nitriding temperature, annealing temperature, ammonia pressure and the duration of the process and the depth of penetration of the counter-body for BH11 and BH21 steels in ion nitriding in ammonia. Based on these models, the technological regimes corresponding to the minimum and maximum penetration of the control in the nitrided layer are determined. It has been shown that after testing the two nitrided steels for wear, the penetration of the counter-body into the nitride zone is different. It depends mainly on the thickness and phase composition of the nitride zone. The highest wear resistance was found after ion nitriding at a nitriding temperature of 550 °C for 10 h and an ammonia pressure in the chamber of 450 Pa. It has been proven that higher wear resistance of nitrided layers occurs when a two-phase (ε + γ') bonded zone is formed when the ε-phase is in a larger amount.

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