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The impact of basic boroaluminizing factors on diffusion layer thickness in low-carbon steels and its mathematical modeling

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Abstract. The paper presents the study of pack boroaluminizing carried out on steel 20 using mathematical planning methods. A mathematical model and nomograms showing the dependence of diffusion layer thickness on basic parameters factors of thermochemical treatment (technological factors such as temperature, treatment time and powder mixture composition) were obtained by means of conducting a full factorial experiment. The degree of impact which technological factors make on the final result and the ranges of their regulation are revealed. The temperature of pack boroaluminizing varied from 900 to 1000 ºC, the duration of the process was 2-4 hours, and the ratio of the treatment components of B_2O_3 / Al was 40/60 to 50/50 wt-%. The metallographic analysis has revealed that the depth of the diffusion layer is from 80 to 260 μm, depending on technological factors.

1. Introduction

Currently in mechanical engineering, to strengthen the surface layer of machine parts, it is possible to effectively use multicomponent thermochemical treatment (TCT) consisting in simultaneous or sequential diffusion saturation with several chemical elements [1,2]. Multicomponent methods of TCT, such as boroaluminizing, allow to increase wear resistance, improve heat resistance, oxidation resistance and a number of other properties of surface layers of machine parts [3-6]. Boroaluminizing is implemented in different ways: in treatment pastes, in powder mixtures of boron and aluminum-containing substances, in liquid and gaseous media.

For a broader practical application of this technology, mathematical models are needed. They generalize experimental data and allow to reliably control the technological process and predict the result of processing. Such models of TCT processes in the form of regression equations or in the form of power functions are presented in [7-10]. They are convenient for calculating output parameters, optimizing technological modes and can be quite accurate (~ 8-10%). To construct a mathematical model of the TCT process, in order to reduce the number of experimental studies, the method of mathematical planning of the experiment was used. The TCT process comprises a fairly large number of factors affecting the output properties of the surface layer. Full factorial experiment method was used to determine the optimal parameters of technological modes and to obtain the dependences of the examined properties from these parameters.

The purpose of this work is to investigate the dependence of the diffusion layer thickness on the temperature, treatment time and saturating mixture composition at pack boroaluminizing. On the basis of the dependence obtained, a mathematical model will be constructed that describes the influence of the main boroaluminizing factors on the diffusion layer thickness.

To achieve the proposed aim, it is necessary to solve the following objectives:

- to create and implement an experiment plan that provides an opportunity to obtain an adequate mathematical model of the boroaluminizing process;
- to carry out a metallographic analysis to estimate the diffusion layers thickness after boroaluminizing;
- to obtain a graphoanalytical description of the variation in the diffusion layer thickness, that will be convenient for selecting modes and predicting the result of the treatment.

2. Materials and methods

The steel 20 was chosen as the object of the research. Boroaluminizing of the steel samples was carried out in a powder mixture with composition of 70% Al2O3 + 30 (x% B2O3 + y% Al) + 0.5% NaF in a container with a fusible gate in isothermal exposure. The thickness of the boroaluminized layers was evaluated on optical microscope “Neophot-21”. To obtain the relationship between treatment parameters and diffusion layer thickness, the method of mathematical planning of the experiment was applied. Data analysis of a priori information, as well as the results of experiments [2] allowed to distinguish three main factors (in code values): X1 - treatment time in hours; X2 – treatment temperature in degrees Celsius; X3 - composition of treatment mixture in percentage ratio of B2O3 and Al. The experiments were carried out according to the full factorial experiment algorithm of type 23, where the number of factors is k = 3, the number of levels is p = 2. To calculate the coefficients, it is necessary to perform N = 8 experiments, the number of repeated experiments is n = 5. The ranges of regulation of the main technological factors are given in Table 1.

Table 1. Technological factors and their variation levels.

Factors	Variation levels			Variation interval
	-1	0	+1	
x_1 – treatment time, h	2	3	4	1
x_2 – temperature, °C	900	950	1000	50
x_3 – B_2O_3/Al ratio, %	40/60	45/55	50/50	5/5

3. Experimental results

Creating a mathematical model comes down to obtaining an equation of the first or second order in the form [10]:

$$Y = b_0 + b_1x_1 + b_2x_2 + ... + b_nx_n + b_{11}x_1^2 + b_{22}x_2^2 + ... + b_{12}x_1x_2 + ... + b_{nm}x_nx_m \quad (1)$$

where Y – calculated boroaluminized layer thickness, μm;
x_1, x_2, x_n, x_m – variation parameters (Table 2);
b_0, b_n, b_{nm} – regression coefficients.
Table 2 shows the planning matrix and averaged results of the performed experiments.

Table 2. Planning matrix of the experiment.

Number of experiment, No	Planning matrix								Average value of boroalumi-nized layer thickness, Yi, μm
	x_0	x_1	x_2	x_3	x_1x_2	x_1x_3	x_2x_3	$x_1x_2x_3$	
1	+1	-1	-1	-1	+1	+1	+1	-1	110.6
2	+1	+1	-1	-1	-1	-1	+1	+1	180.8

3	+1	-1	+1	-1	-1	+1	-1	+1	160.2
4	+1	+1	+1	-1	+1	-1	-1	-1	259.8
5	+1	-1	-1	+1	+1	-1	-1	+1	53.6
6	+1	+1	-1	+1	-1	+1	-1	-1	80
7	+1	-1	+1	+1	-1	-1	+1	-1	209.8
8	+1	+1	+1	+1	+1	+1	+1	+1	244.4

Experiments and processing of results allowed to obtain an equation for calculating a boroaluminized layer thickness in the given interval of main factors variation: treatment temperature, treatment time and composition of saturating mixture:

$$Y = 162{,}4 + 28{,}85x_1 + 56{,}15x_2 - 15{,}45x_3 + 4{,}7x_1x_2 - 13{,}6x_1x_3 + 24x_2x_3 \quad (2)$$

The obtained mathematical model was tested for adequacy by Student's and Fisher's criteria. The procedures of evaluating the coefficients of mathematical model, verification of its adequacy and the statistical analysis of accuracy was calculated according to sources [8,9,10].

Coefficients significance examination is implemented by comparing the absolute coefficient value with the confidence interval. To do this, is to be calculate the variance of reproducibility by following formula:

$$S^2_{\{y\}} = \frac{\sum_{1}^{N} S_i^2}{N} = 14{,}25 \quad (3)$$

Next, is to be find the confidence interval by formula:

$$\Delta b_i = \pm \frac{t \cdot S_{\{y\}}}{\sqrt{N}} = 3{,}746 \quad (4)$$

where t is the tabular value of criterion at the accepted significance level and the number of degrees of freedom f with which the variance S_y^2 was determined. The value of t for five repeated experiments and the confidence probability of 0.95 is 2.78 [10]. Coefficients significance evaluation of the model with the use of Student's t-test showed that the coefficient b_{123} equal to 2.65 can be considered not significant.

The adequacy dispersion, characterizing the empirical values scattering relative to the calculated values Y, is to be found by the formula:

$$S^2_{a\partial} = \frac{\sum_{1}^{N} (Y_{iu} - Y_i)^2}{f} = \frac{\sum_{1}^{N} \Delta Y_i^2}{f} = 67{,}86 \quad (5)$$

where Y_{iu} is the parameter arithmetic mean in the i-th experiment; Yi - value of the investigated parameter, calculated by the model for the i-th experiment conditions; f is the number of degrees of freedom equal to $N-(k+1)$; where k is the number of factors.

The adequacy of the mathematical model is to be determined by means of the Fisher’s criteria:

$$F_{расч} = \frac{S^2_{ад}}{S^2_{\{y\}}} = 4,672 \quad (6)$$

Mathematical model is considered adequate as long as $F_{calc} \leq F_{table}$.

The adequacy dispertion calculation is given in Table 3, where the number of degrees of freedom: $f_5 = f_7 = 5 - 1 = 4$. The results of the model validation on adequacy are shown in Table 4.

Table 3. Adequacy dispertion.

No. of experiment	Boroaluminized layer thickness, Y, μm		ΔY	ΔY^2
	Experimantal values	Calculated values		
1	110.6	107.95	2.65	7.0225
2	180.8	183.45	-2.65	7.0225
3	160.2	162.85	-2.65	7.0225
4	259.8	247.75	12.05	145.2025
5	53.6	56.25	-2.65	7.0225
6	80	86.75	-6.75	45.5625
7	209.8	216.55	-6.75	45.5625
8	244.4	247.05	-2.65	7.0225
	ΣΔY2			271.44

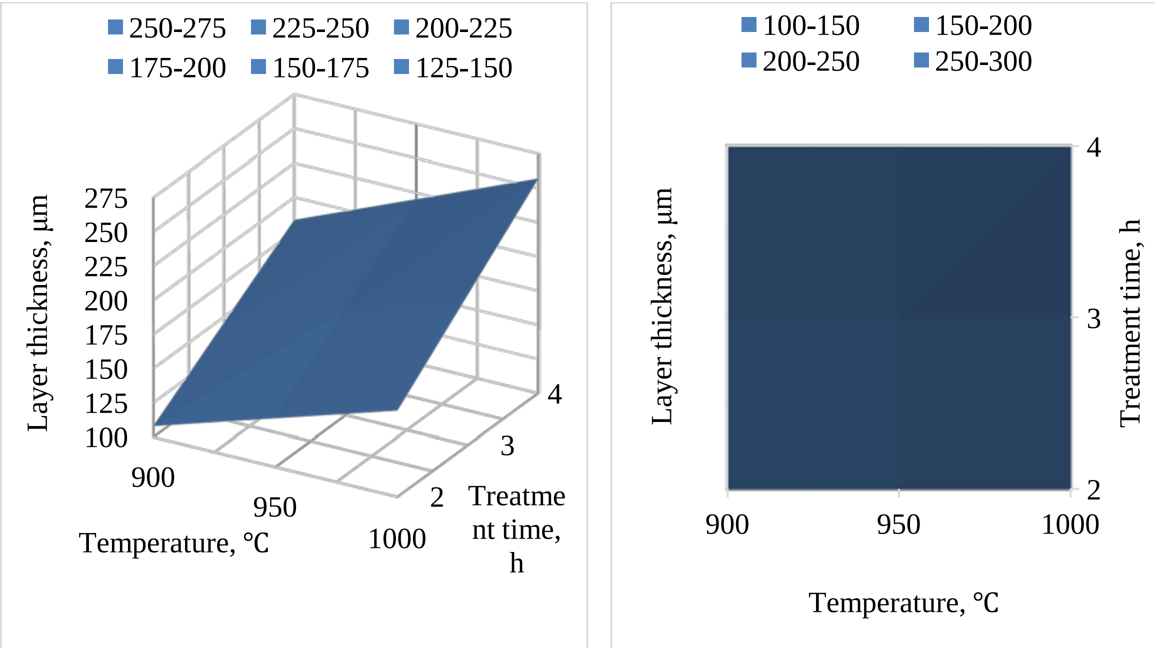
Table 4. Prove of model adequacy calculation.

$S^2_{\{y\}}$	$S^2_{ад}$	F_{calc}	F_{table}
14.525	67.86	4.6719	6.4
$F_{calc} \leq F_{table}$, the model is adequate			

4. Graphical processing of experimental results and discussion

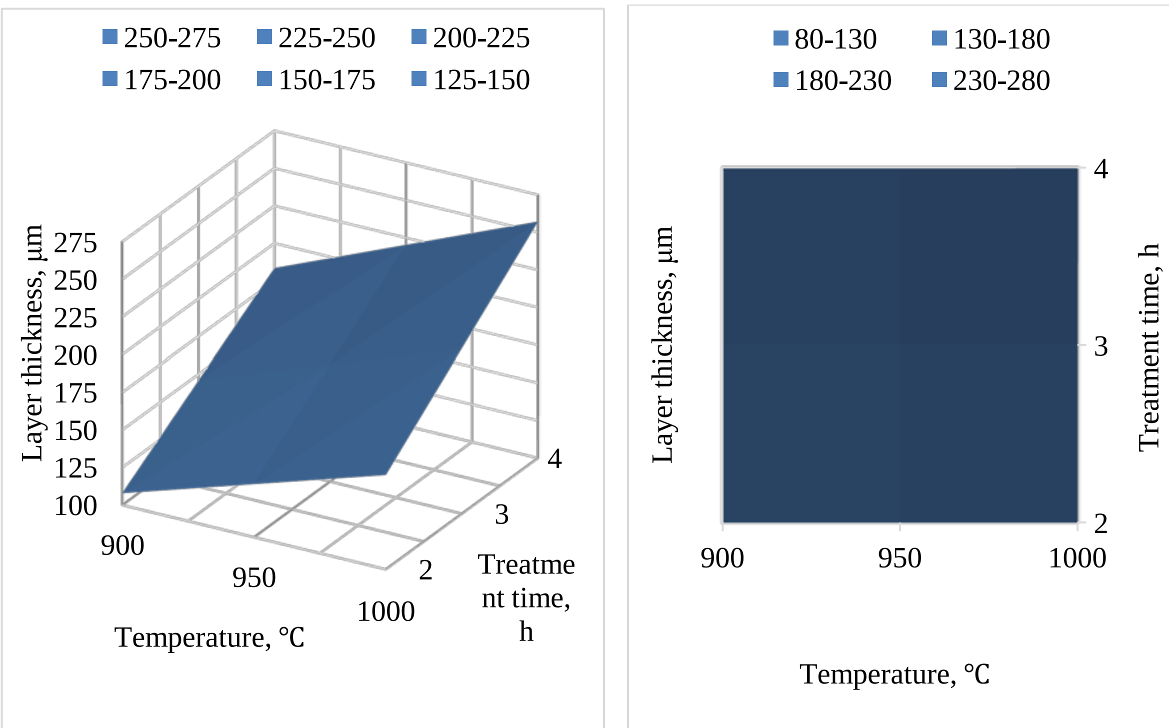
Surfaces of the response and nomograms, depending on the technological factors of the boroaluminizing process, are shown in Figs. 1-3. As follows from the results of the investigation and modeling, the optimal values of the boroaluminized layer thickness correspond to the region of maximum values of the duration and temperature of the process. The most practical is a graphoanalytical representation that will allow make selection and predict the diffusion layer thickness based on two technological factors: the treatment temperature and time.

The composition of treatment mixture (the third technological factor) is recommended to be selected depending on the required surface properties. For higher oxidation resistance at high temperatures, a predominantly aluminized layer is required, and for enhanced wear resistance, a predominant boronized layer is needed [3]. The nomograms shown in Figs. 1b, 2b and 3b are the cross sections of the response surface at a given level of output variables. Such a graphoanalytical description allows one to predict the boroaluminized layer thickness under the combined influence of the temperature and the time of the TCT process for various composition the saturating mixture.



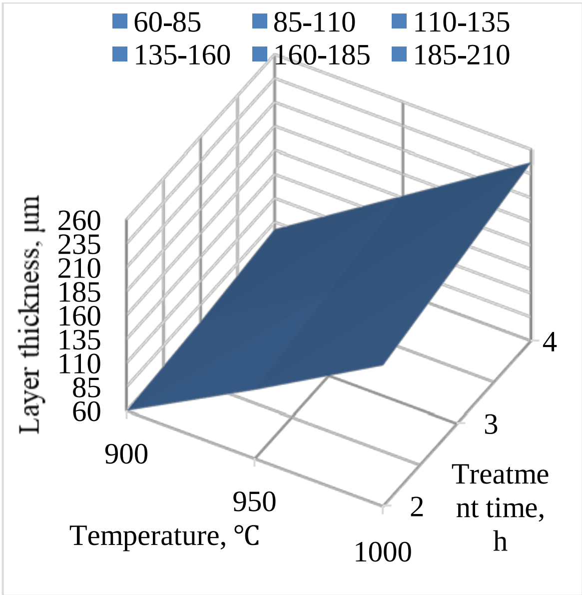
(a) (b)

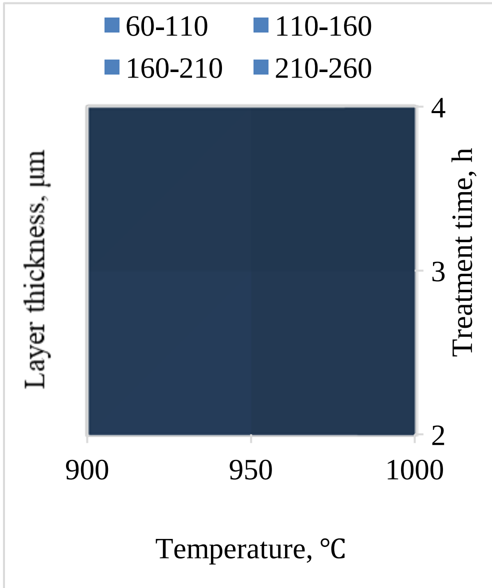
Figure 1. Response surfaces (a) and nomographs (b) in the factor space of the boroaluminized layer thickness variation depending on the treatment temperature and time. The composition of the saturating mixture 40% B_2O_3 + 60% Al (lower level).



(a) (b)

Figure 2. Response surfaces (a) and nomographs (b) in the factor space of the boroaluminized layer thickness variation depending on the treatment temperature and time. The composition of the saturating mixture 45% B_2O_3 + 55% Al (zero level).

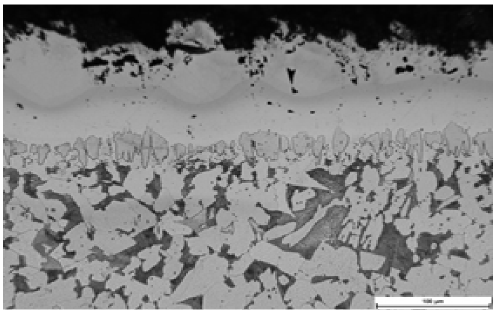




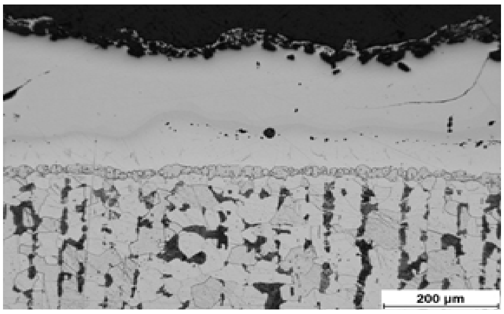
(a) (b)

Figure 3. Response surfaces (a) and nomographs (b) in the factor space of the boroaluminized layer thickness variation depending on the treatment temperature and time. The composition of the saturating mixture 50% B_2O_3 + 50% Al (upper level).

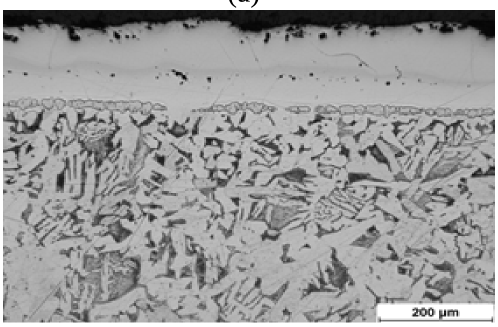
Figure 4 shows the microstructures of boroaluminized layers.



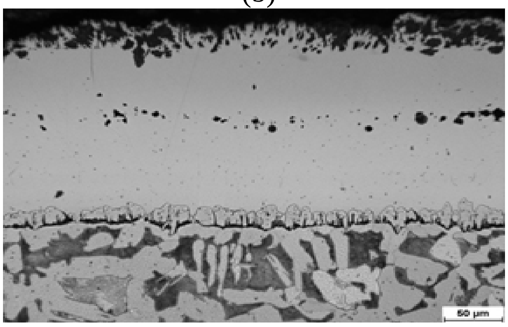
(a)



(b)



(c)



(d)

Figure 4. Microstructures of boroaluminized layers on steel 20 after treatment in saturating mixture with percentage ratio of 40%B_2O_3 + 60%Al: a) 900 °C, 2 hours, average layer thickness is 110 µm; b) 900 °C, 4 hours, average layer thickness is 160 µm; c) 1000 °C, 2 hours, average layer thickness is 180 µm; d) 1000 °C, 4 hours, average layer thickness is 260 µm.

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5. Conclusions

- Implemented experiments and processing of the obtained results allowed to obtain an adequate mathematical model of the dependence of the boroaluminized layer thickness on the values of the temperature, the treatment time and the composition of the saturating mixture.
- The obtained graphoanalytical description in the form of response surfaces and nomograms is a convenient tool for predicting the diffusion layer thickness.

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