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RESEARCH ARTICLE

Analysis of boride layer thickness of borided AISI 430 by response surface methodology

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ABSTRACT

The boriding process is a thermochemical surface treatment which can be applied to many iron and non-ferrous materials and improves the properties of the material such as hardness, wear resistance. In the present study, the layer thickness values of the boronized AISI 430 material were optimized using the Response Surface Methodology. Mathematical model was constructed using parameters such as temperature and time and the results were analyzed comparatively. As a result of the analysis, the optimum layer thickness value for AISI 430 material was obtained as 39.0183 µm for 1000 ºC and 5.9h and it was determined that the boriding temperature and time are effective on the boride layer formation process of AISI 430 material. Finally, the Response Surface Methodology and Face Centered Central Composite Design have been effectively applied to the boriding process.

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1. Introduction

Surface treatments are applied to overcome the problems of exposed materials such as corrosion, abrasion, oxidation [1-2]. The surface properties of machine elements and tools can be improved by diffusing the atoms of various materials. Carbonization, nitriding, chroming and boriding processes are diffusion methods used to improve the surface properties of materials [3].

Boriding is one of the most well-known thermochemical surface hardening processes that provide features such as high surface hardness, corrosion and abrasion resistance [4-6]. Approximately in 800 – 1100 ºC, process is applied for 2 - 8 hours [7]. Boriding can be applied to materials using various methods, such as solid, liquid, and gas [8]. The solid boronizing method used in the study is a simple and economical method compared to other methods. AISI 430 Stainless Steel materials are used in a variety of industries. AISI 430 is especially used in the automotive industry for machine parts such as exhaust manifolds, turbochargers and catalytic converters [9]. Optimization studies of the boriding process are limited in the literature [10-13].

Genel et al. [10] studied that the boride layer properties of the boronized AISI W1 material by solid boronizing method were modeled mathematically by Artificial Neural Networks (ANN) and the boride layer thickness was estimated to be approximately 95%. They concluded that borided layer thickness increases with boriding time for each process temperature. Besides, they found that surface hardness of layer increased approximately 6 times compared to non-borided material.

Arguellas - Ojeda et al. [11] investigated the hardness of ASTM F-75 alloy, which borided by paste method. They performed process optimization through Response Surface Methodology (RSM) and determined the model effects of process factors. They developed response surface equation to analyze the effect of values on borided layer hardness. Developed model showed that processing temperature has significant whereas processing time has no significant effect on the boride surface hardness. Under these conditions, they determined that maximum hardness value can be obtained by RSM.

Chen et al. [12] reported that they optimized boride layer depths of the boronized Cr12MoV material by Response Surface Methodology. As a result of obtained values, they determined that the depth of the boride layer increased with the increasing temperature in the process and applied heat treatment processes such as quenching and tempering to investigate their impact on wear resistance.

Kayalı et al. [13] applied boriding process in three different temperature and time parameters using box boriding method to AISI 52100 material. Afterward, they analyzed the wear behaviors of the boronized AISI 52100 material by the Taguchi Method and determined the optimum parameters. According to Taguchi Analysis, the most effective parameter was boriding temperature and wear resistance of AISI 52100 increased as the boriding temperature and time increases. Besides, they found wear load and sliding rate effect on surface wear resistance as the second and third effective parameter.

In order to obtain boride layer thickness, the process parameters must be selected appropriately. In this study; the layer thicknesses of the boronized AISI 430 material were optimized through the Face Centered Central Composite Design (FCCD) using the Response Surface Method. Due to the fact that it is difficult to apply the AISI 430 containing high alloy elements, the thickness of boride layer has been maximized by using RSM. In the RSM model, temperature and time were determined as input, and layer thicknesses were determined as output. The parameters affecting the model and the results are given comparatively. In light of this study, appropriate technique can be determined and used in industrial applications in comparison with other optimization techniques for boriding heat treatment. Besides, the effect of heat treatment such as boriding, chromizing and nitriding on the mechanical properties of material can be optimized by using RSM. Due to the high cost of heat treatment applications, the importance of process optimization is high. The high temperature and process materials required for the boriding process make the optimization work inevitable

Figure 1. Heat treatment furnace where boriding process is performed.

2. Experimental work

2.1. Boriding process conditions

The AISI 430 test specimens to be used in the work were cut to a diameter of 20 x 20 mm and made ready for boriding. The method chosen for the boriding process is the box boriding method. After filling the supplied B_4C + SiC + KBF_4 powders into the stainless steel box, the metallographically prepared samples were embedded in the (B_4C + SiC + KBF_4) powders.

The boriding process was carried out in the electric controlled furnace by increasing gradually to the temperatures determined in 2-4-6 hours at 850-925-1000ºC temperatures.

Subsequently, the samples removed from the furnace were left to cool down in the air. Finally, AISI 430 samples were ground to the 1200 grid level by SiC paper and polished, then made ready for optical microstructural examination using (100ml ethanol + 5ml HCl + 1g picric acid) etching. Layer thicknesses of boronized AISI 430 materials were measured with a Leica Optical Microscope by the aid of a microscope-assembled tool.

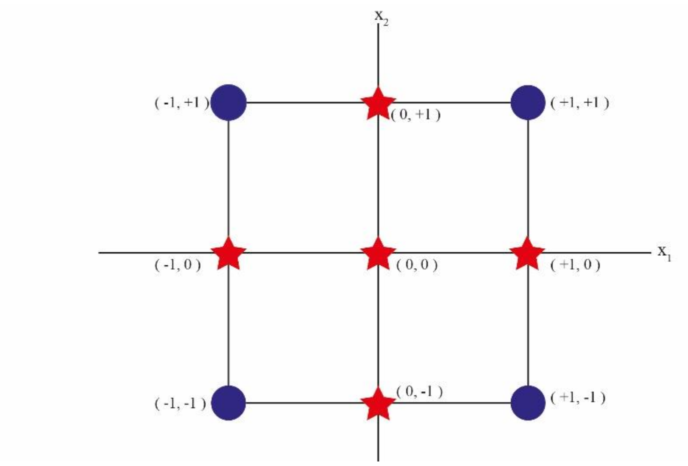

Figure 2. Central composite design for Face Centered Central Composite Model (FCCD).

Table 1. Coded and uncoded level of factors.

Factors	Coded and Uncoded Levels		
	-1 (Low)	0 (Medium)	+1 (High)
Temperature (ºC)	850 ºC	925ºC	1000ºC
Time (Hour)	2h	4h	6h

2.2. Statistical design of borided AISI 430 by response surface methodology

In this study, the layer thicknesses of boronized AISI 430 were statistically examined by using FCCD. The FCCD consists of axial points located on the cube five-sided surface. Central Composite Design (CCD) is used to develop experimental design in RSM [14-17]. The interaction between the inputs and the experimental variables were examined and the statistical results were presented comparatively. Compared to other optimization techniques such as Taguchi Method, the RSM gives optimum results with decimal system of factor levels, while in Taguchi analysis, the best combination can be obtained for given factors [18].

In the work, three levels were chosen for the factors used as input when creating the experimental design. These values were coded as -1, 0, +1. The factors used in the experimental design and the coded levels are shown in Table 1. The MINITAB 16 package program was used to determine the results of the mathematical models of the experimental design. Graphs of the results obtained from the program were also obtained through the MINITAB 16 package software.

3. Results and discussion

Layer thicknesses of boronized AISI 430 stainless steel were modeled mathematically using the FCCD method at three different temperatures and at three different time durations.

Equation 1 describes the mathematical model on the results (Y) of the relevant factors used in the design of the experiment.

$$Y = \beta_0 + \sum_{i=1}^{n} \beta_i X_i + \sum_{i=1}^{n} \beta_{ii} {X_i}^2 + \sum_{i=1}^{n} \beta_{ij} X_i X_j + e \quad (1)$$

According to Eq. (1); Y value is defined as the response value, and x_i and x_j are the coded values of the factors.

β_0, β_i, β_{ii}, β_{ij}, represent the regression coefficient, i and j are the linear and quadratic coefficients, respectively. e is the residual experimental error.

ANOVA (Analysis of Variance) was performed to show the significance and interaction of the factors used in the mathematical model.

The statistical analysis results are shown in Table 2 and Table 3.

Table 2. Face centered composite design of two factors for coded, uncoded and response (boride layer thickness).

No.	Coded Values T	Coded Values t	Uncoded Values Temperature (ºC)	Uncoded Values Time (Hour)	Boride Thickness *Y* (µm)
1	-1	-1	850	2	10
2	1	-1	1000	2	21
3	-1	+1	850	6	17
4	+1	+1	1000	6	38
5	-1	0	850	4	12
6	+1	0	1000	4	32
7	0	-1	925	2	19
8	0	+1	925	6	33
9	0	0	925	4	25
10	0	0	925	4	26
11	0	0	925	4	26
12	0	0	925	4	25
13	0	0	925	4	27

Y, Response (Boride Layer Thickness) = 25.2069 + 8.6667T + 6.3333t - 4.1379T^2 - 0.1379t^2 + 2.500Tt (2)

Table 3. Analysis of variance for boride layer thickness.

Source	Sum of Squares	Degrees of Freedom	Mean Square	*F*	*p* - value
Regression	773.118	5	154.624	110.39	0.000
Linear	691.333	2	345.667	246.79	0.000
T	450.667	1	450.667	321.75	0.000
t	240.667	1	240.667	171.82	0.000
Square	56.785	2	56.785	20.27	0.001
T x T	56.733	1	47.291	33.76	0.001
t x t	0.053	1	0.053	0.04	0.852
Interaction	25.000	1	25.000	17.85	0.004
T x t	25.000	1	25.000	17.85	0.004
Residual Error	9.805	7	1.401		
Lack of Fit	7.005	3	2.335	3.34	0.137
Total	782.923	12			

R-Sq = 98.75%, R-Sq (pred.) = 90.51%, R-Sq (adj.)= 97.85

As a result of statistical analyzes; R^2 (coefficient of determinant) and R^2-adj (adjusted R^2 value) were found to be 98.75 and 97.85, respectively. R^2 (coefficient of determination) indicates that 98.75% of the model is affected from mathematical model whereas, R^2-adj (adjusted R^2 value) is the value that is calculated after subtracting insignificant values from the mathematical model. If the p value obtained from the ANOVA is less than 0.05, the model for that parameter is significant.

When Table 3 is examined, the p values are important for the main factors. In other words, all the main factors are important for the layer thickness of the boronized AISI 430 material.

In mathematical model, Temperature (T), Time (t), [Temperature (T) x Temperature (T)], [Temperature (T) x Time (t)] is significant, while [Time (t) x Time (t)] is insignificant.

Equation 2 represents the mathematical model which was developed for the boride layer thickness (Y) of boronized AISI 430 material.

In order to reveal the effect of the factors on the results, 3D and 2D interaction graphs were created using the MINITAB 16 package software.

Contour plots for the interaction effects of factors (time and temperature) as 2D and 3D are given in Figure 3.a. and Figure 3.b. Figure 3.a. and Figure 3.b. show that the temperature (T) and time (t) factors have a positive effect on the boride layer. It is also reported in the previous boriding studies that the temperature and time parameters are effective on the formation of the boride layer and the increase in the layer thickness is generally accompanied by increasing temperature and time [1,5].

The correlation graph of the experimental and predicted results is shown in Figure 5. In Figure 5, it is clearly shown that experimental values and predicted values are close to line. This case indicates high correlation between the values.

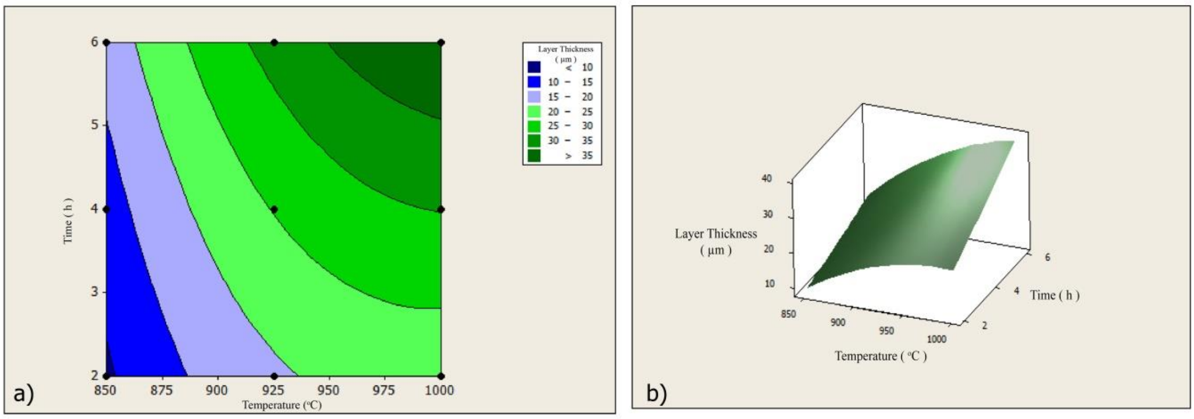

Figure 3. a) Contour plot for borided layer thickness. **b)** Response surface plot for borided layer thickness.

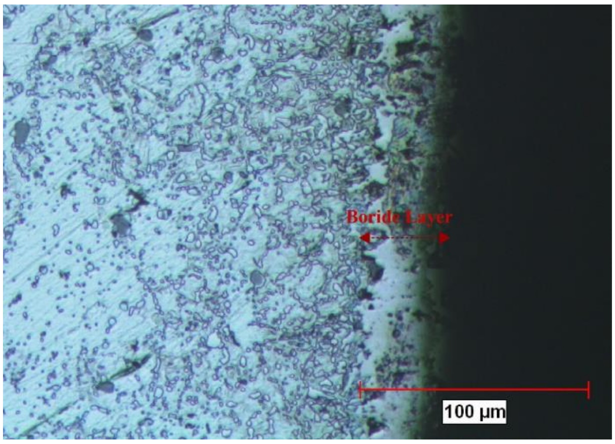

Figure 4. Optical microstructure image of borided AISI 430 material.

Furthermore, it has been stated in literature [19-21] that the boride layer thickness value of the target material during the boronizing process may vary according to the boron source content and the alloy content of the target material as well as the temperature and time.

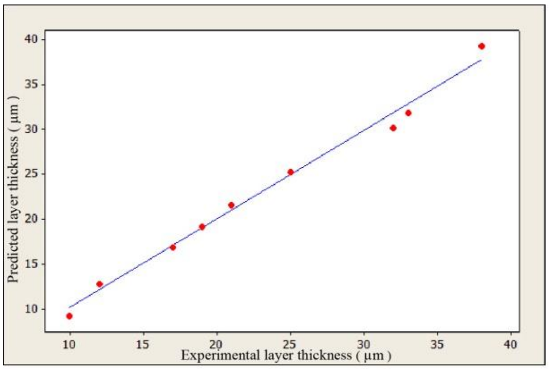

Figure 5. The correlation graph of the experimental and predicted results.

Optical microstructure images of boronized AISI 430 are given in Figure 4. According to Figure 4; the boride layer of the boronized AISI 430 material was found to be a planar structure. As seen in Figure 4, there is no porosity or discontinuity in the boride layer morphology. The cause of this condition is considered to be made of properly the boriding process. When the values of layer thicknesses of boronized AISI 430 were examined, it was determined that the layer thickness values increased parabolically as the temperature and time increased.

4. Conclusion

In this study, it has been showed that the RSM Method can be effectively applied to the boriding process of AISI 430 material.

Layer thickness values of boronized AISI 430 material using box boriding method have been experimentally successfully designed using temperature and time parameters by FCCD.

As the result of ANOVA analysis; R^2 and R^2 (adj) values of 98.75 and 97.85 were found.

Except for the factor (t x t) (p value: 0.852), all the main factors in the mathematical model for the layer thickness of the boronized AISI 430 are significant.

According to analysis results; optimum conditions for the boride layer thickness of AISI 430 were obtained at 1000 ºC and 5.9 hours, respectively.

According to the results of this input, the optimum value of the boride layer thickness is 39.0183 µm.

Consequently, the generated mathematical model has proven to be able to explain the boriding process at a high rate.

Because of the costly surface treatment methods such as the boriding process, successful implementation of the optimization operation is thought to be a significant contribution to cost and time.

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