International Journal of Engineering Research & Technology (IJERT)
ISSN: 2278-0181
Vol. 3 Issue 9, September- 2014

Modeling of Pack-Carburizing Route by General Factorial Design of Experiment

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Abstract—**This work modeled the influence of Na_2CO_3 as an activator material in the pack-carburizing process of 1.5920 steel by General Factorial Design of Experiment (GFDE). Four different carburizing mixtures containing 0, 5, 10 and 15 wt. % of Na_2CO_3 at 16 experiments have been used. The samples were carburized at 925°C for different time of 3, 5, 8 and 12 hrs. The Effective Case Depth (ECD) of treated samples was measured using a micro-hardness test. The activator content and carburizing time were considered as model factors. The optimal conditions to attain the maximum ECD were predicted by GFDE. The results indicated that by using activator amount of 11.5 wt. %, maximum ECD could be achieved, regardless of the carburizing time. The reasons for declining of ECD corresponding to the activator amount beyond the 11.5 wt. % were also discussed.**

Keywords— ***Pack-Carburizing; Activator; 1.5920 steel; Modeling; General factorial design of experiment.***

I. INTRODUCTION

Surface hardening is a very important process for industrial applications. Machine components such as shafts, gears and cams often require a very hard surface that can resist wear and a soft, tough core that can withstand the impact stresses which occur during operation. An established method for the production of such a combination of hard case and soft, tough core is case hardening of steels through carburizing and quenching [1-3]. Carburizing is the addition of carbon to the surface of low carbon steels at temperatures generally between 850-950 °C (1560-1740 °F), at which austenite, with its high solubility for carbon, is the stable crystal structure. Hardening is accomplished when the high-carbon surface layer is quenched to form martensite [1,2]. As a result of this process a high-carbon martensitic case with good wear [4,5] and fatigue resistance [6,7] is superimposed on a tough low-carbon steel core.

Carburizing steels for case hardening usually have base-carbon contents of about 0.2 wt.%, with the carbon content of the carburized layer generally being controlled between 0.8 and 1 wt.%. However, surface carbon is often limited to 0.9 wt.%, because so high carbon content can result in retained austenite and brittle martensite [6,8]. It has been reported [9] that the Martensite Finish Temperature (M_f) for carbon content greater than 0.65 wt.% value is below room temperature.

It is well documented that many factors, such as time, temperature, and surface carbon influenced the final microstructure and properties of treated samples [10-15]. In contrast to the gas and liquid carburizing, solid carburizing is a minor commercial process. It requires more processing time. Obtaining greater case depths by increasing time cycles is costly due to increasing energy consumption [16]. Case depth can be increased exponentially by increasing the carburizing temperature, but this approach is also problematic in economic sense [17]. It has been reported that adding of some rock minerals [10, 18-21] or Rare Earths (RE) [22,23] in carburizer can accelerate the carburizing process. For example, Ogo et al. [18] observed that there was significant increase in the carburization rate of mild steel by the addition of river clam shell (mainly contains $CaCO_3$) to charcoal. Jimenez et al. [19] reported that addition of carbonates ($BaCO_3$ and Na_2CO_3) to the metallurgical coke gave rise to an increase in the carburization rate and case depth which allowed the achievement of the required carbon concentration profiles more efficiently. From the industrial point of view, it is essential to find out the best combination of carburizing parameters to attain the maximum case depth.

One of the most common and classical approaches employed by many experimenters is One-Factor-At-a-Time (OFAT), in which one factor is varied while all other variables or factors in the experiment are fixed. The success of this approach depends on guesswork, luck, experience and intuition. Moreover, this type of experimentation requires large resources to obtain a limited amount of information about the process [24-29]. OFAT experiments often are unreliable, inefficient, time consuming [30] and may yield false optimum condition for the process. The major disadvantage of the OFAT strategy is that it fails to consider any possible interaction between the factors [24-29]. An interaction is the failure of the one factor to produce the same effect on the response at different levels of another factor [27].

Statistical thinking and statistical methods play an important role in planning, conducting, analyzing and interpreting data in engineering experiments. When several variables influence a certain characteristic of a product, the best strategy is to design an experiment so that valid, reliable and sound conclusions can be drawn effectively, efficiently and economically [26,27,29]. This is an experimental strategy in which factors are varied together, instead of one at a time [27,29].

It is widely accepted that the most commonly used experimental designs in experimentation are General Factorial Design of Experiment (GFDE). GFDE would enable an experimenter to study the joint effect of the factors on a response. In the present work, using GFED, an appropriate experimental procedure was designed to optimize the Effective Case Depth (ECD) of 1.5920 steel in the presence of activator material (i.e. Na_2CO_3). A model was then developed for predicting the ECD of this steel.

II. EXPERIMENTAL PROCEDURE

A. Exprimental

The chemical composition of 1.5920 steel used in this work is listed in Table 1. To facilitate experimental works such as carburizing process and micro-hardness test, steel specimens were cut in the form of cubes with 3 cm in dimensions and thoroughly washed in acetone and allowed to dry. Pack carburizing boxes with 10×10×10 cm^3 were made using low carbon steel sheets, whose thickness was 0.5 cm. An electric muffle furnace made in Iran was used.

TABLE I. CHEMICAL COMPOSITION OF 1.5920 STEEL

Elements	wt. %	Elements	wt. %
C	0.210	Si	0.209
Mn	0.900	Ni	1.823
Cr	0.911	Cu	0.149
Mo	0.027	Al	0.024
P	0.011	S	0.004
V	0.004	Fe	Rem.

To prepare carburizing mixture, the used activator material (i.e. Na_2CO_3) was solved in hot water and then graphite powder was added to the solution. By agitation, the mixture was converted to glass form. Then, the mixture was subjected to sunlight until humidity was totally removed. In the present research, the mixtures were prepared with 0, 5, 10 and 15 wt. % pure Na_2CO_3. A single specimen was placed in the center of each box and the remaining space was filled carefully with the carburizing mixture. The box was covered with a lid and sealed with the fireclay to prevent air infiltration into the box during carburization. The box containing the test specimen was then placed in the central zone of the furnace, which was already at the required temperature of 925°C. Carburizing durations of 3, 5, 8 and 12 hrs were also used. At the end of each test, the box was taken out of the furnace and the sample was quenched in oil. Each test was repeated for at least three times. Vickers micro-hardness testing machine (model MHT.1; No: 8331) made by Matsuzawa Seiki Co Ltd of Japan was used. The carburized specimens were cut from the central region. The samples were then prepared for the micro-hardness test. The micro-hardness test was performed employing a Knoop indenter at every 0.1 mm from the edge of the samples to the center according to ASTM E384-99 standard. ECD was defined as the distance below the surface where the hardness was equal to 550 VHN [31,32].

B. Design of experiment

After a brainstorming session, two factors and their levels were chosen as independent input factors: Carburizing time and weight percent of Na_2CO_3. GFDE was selected as the experimental design method to identify the factors which have the sequence of significance of each effect. Factors and their levels are shown in Table 2. The experiments were performed in random order to ensure that uncontrolled factors did not influence the results [30,33,34]. The response variable of interest was to reach the maximum ECD. The design and statistical analysis of experiments was done by Design-Expert 7 (State-Ease, Inc., Trial version) software.

TABLE II. SELECTED FACTORS AND THEIR LEVELS

Factor	Symbol	Unit	Level 1	Level 2	Level 3	Level 4
Carburizing time	A	hrs	3	5	8	12
Na_2CO_3	B	wt. %	0	5	10	15

III. RESULTS AND DISCUSSION

Fig. 1 represents the hardness profiles as a function of distance from edge at various carburizing times and different amounts of the activator material. The values of ECD for different conditions extracted from these graphs are given in Table 3.

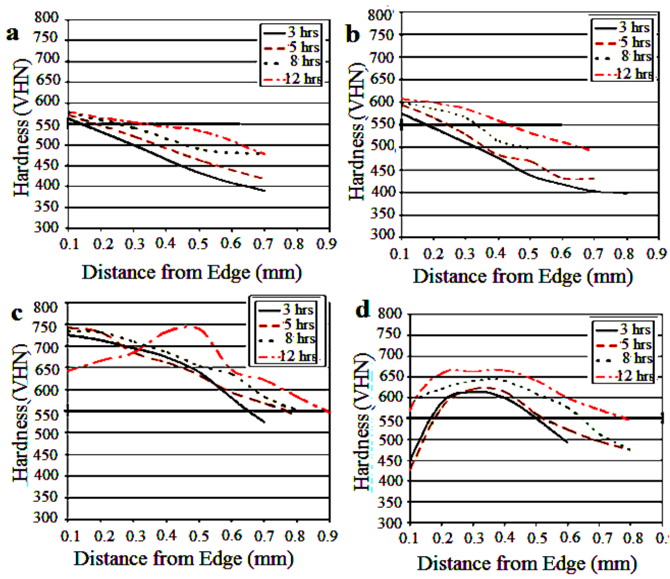

Fig. 1. The hardness-distance curves for carburized samples with different amounts of energizer material (a) 0, (b) 5, (c) 10 and (d) 15 wt.%

The results of the GFDE are presented in Analysis of Variance (ANOVA) table (Table 4) with a Confidence Interval (CI) of 99% for model. In statistics, CI is a kind of interval estimate of a population parameter and is used to indicate the reliability of an estimate. The level of confidence of CI would indicate the probability that the confidence range captures this true population parameter given a distribution of samples [26-28]. By considering half normal plot and normal plot (not show here), two main effects and one two-factor interactions all with CI over 90% were selected as significant factors for modeling. The effect of a factor is defined as the change in response produced by change in level of factor. This is frequently called a main effect because it refers to primary factors of interest in the experiment [29]. ANOVA results for ECD shows a significant model with adequate precision of 65.105.

TABLE III. GFED, EXPERIMENTAL SEQUENCES AND EXPERIMENTAL RESULTS

Standard Order	Run Order	Factor A Carburizing time (hrs)	Factor B Na_2CO_3 wt.%	Response ECD (mm)
1	9	3	0	0.14
2	4	5	0	0.19
3	16	8	0	0.25
4	7	12	0	0.32
5	12	3	5	0.18
6	2	5	5	0.24
7	14	8	5	0.32
8	7	12	5	0.42
9	8	3	10	0.65
10	1	5	10	0.76
11	5	8	10	0.80
12	3	12	10	0.89
13	11	3	15	0.47
14	6	5	15	0.57
15	15	8	15	0.64
16	13	12	15	0.74

TABLE IV. ANOVA TABLE WITH CI OF OVER 90% FOR FACTORS

Source	Sum of squares	Degree of freedom	Mean square	F value	P-value Prob> F
Model	0.93	5	0.19	504.66	<0.0001
A	0.11	1	0.11	309.35	<0.0001
B	0.44	1	0.44	1205.48	<0.0001
AB	0.0014	1	0.0014	3.83	0.0787
B^2	0.055	1	0.055	149.86	<0.0001
B^3	0.023	1	0.023	627.17	<0.0001
Residual	0.0036	10	0.00036	-----	
Cor Total	0.93	15	----		

Adequate precision compares the range of the predicted values at the design points to the average prediction error. On the other hand, adequate precision measures the signal to noise ratio and a ratio greater than 4 is desirable [25]. Here, the value of the ratio is greater than 4, and then it represents the adequate model for predicting the results within design space without doing any further experiments. The analysis of the regression coefficients of the polynomial model describing the relationship between the response and factors, in terms of coded forms, is shown as Eq. 1.

$$ECD(mm) = 0.56 + 0.11t + 0.8E + 0.017t.E - 0.13E^2 - 0.60E^3 \quad (1)$$

In this model, t and E are time (hrs) and Na_2CO_3 wt. %, respectively. To convert from actual units to coded units the following equation (Eq. 2) must be used.

$$X_{coded} = \frac{X_{Actual} - \overline{X}}{(X_{Hi} - X_{Low})/2} \quad (2)$$

Where $X_{Actual}, \overline{X}, X_{Hi}, X_{Low}$ are actual, mean, higher and lower values of the levels [25].

Sum of Squares (SS) of each factor quantifies its importance in the process and as the value of the SS increases the significance of the corresponding factor in the undergoing process also increases. As shown in ANOVA table (Table 4), the effect of factor "B" is the strongest and then B^3, A, B^2 and interaction of A and B (AB), respectively. If we consider the model equation in actual terms, one can found that effect of A, AB and B^2 are positive (synergistic effect). B and B^3 have negative (antagonism) effect on ECD. For increasing ECD, the positive effect should be ascended and negative effect should be descended.

The quality of fittings of the equations was expressed by the coefficient of regression "Adjusted R-squared" or in better way by "Predicted R-squared". The "Adjusted R-squared" values indicate variability in the observed response values which can be explained by the experimental factors and their interactions. The "Predicted R-Squared" and "Adjusted R-Squared" values are closer to 1, the better fitting is achieved [35]. The "Predicted R-Squared" of 0.9896 is in reasonable agreement with the "Adjusted R-Squared" of 0.9941. The Model F-value of 504.66 implies that the model is significant (F_{model} = 504.66 >> F_{table} (5,10) = 10.48) and there is only a 0.01% chance that a "Model F-Value" could occur due to noise. F-Value is the test for comparing the variance associated with that term with the residual variance. It is the mean square for a term divided by the mean square for the residual. This term should be as large as possible [25]. Tables of F-value (a,b) for different confidence intervals exist in statistical references [24], where, the first number in parenthesis is the parameter or model degree of freedom and the second one is error's degree of freedom. To categorize the parameter or the model as a significant value, calculated F-value must be more than its value in the statistical tables. If the calculated value of F is greater than that in the F table at a specified probability level, a statistically significant factor or interaction will be obtained [27]. This model (Eq. 1) can be used to navigate the design space. The relationship between the actual and predicted values that are shown in Fig. 2 is a confirmation for navigation power of the model. As can be seen in Fig. 2, level 2 and 3 of the factor B resulted in higher and less scattered data than other levels. The perturbation plots (not shown here) also confirm these results.

Fig. 3 shows the 3D plot of ECD, as a function of time and Na_2CO_3 wt. %. The surface plot allowed the whole range of conditions to be explored, including the combinations that were not experimentally demonstrated. A greater ECD, in practice, may follow a longer analysis time, which the results show that is not very important factor in our work. In this work, the optimum condition was achieved at Na_2CO_3 content of 11.5 wt. %., and in this range, time had not so important effect.

The confirmation experiments were conducted at A: 12 hrs and B: 11.5 wt. % for 3 times. From these experiments, the amount of ECD was obtained equal to 0.96 mm. If the average of the results of the confirmation is within the limits of the CI, then the significant factors as well as the appropriate levels for obtaining the desired results are properly chosen [26-29]. The predicted range of ECD is 0.98 ± 0.05 mm, then the response (ECD = 0.96 ± 0.01 mm) is in 99% CI range and this model can be used to navigate within the design space.

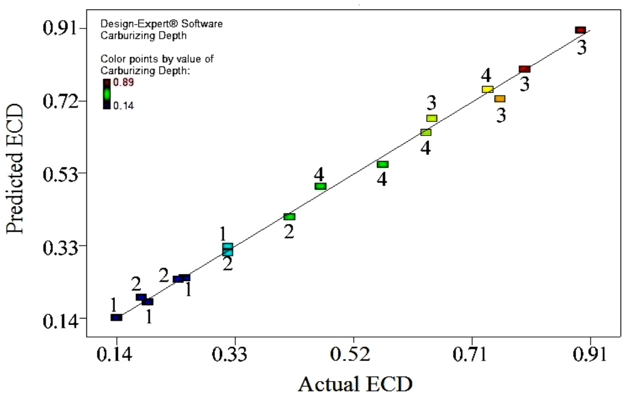

Fig. 2. Predicted vs. actual values. Numbers indicate the level of factor B. For example, number “2” indicates that the amount of factor B is 5 wt.%.

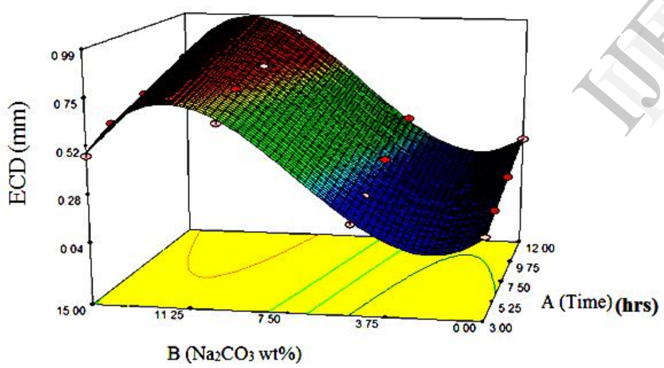

Fig. 3. Graphical representation of the ECD as a function of carburizing time) and Na_2CO_3 wt. %.

The carburizing pack contains some air. The oxygen in the pack's air combines with carbon at elevated temperatures and produces CO_2 (reaction 3). Then CO_2 reacts with carbon atoms present in the pack, and CO is produced (reaction 4). CO decomposes on the steel surface into atomic carbon and CO_2 (reaction 5). The atomic carbon (C_{atom}) is quickly absorbed at the metal surface and diffuses into the metal. Again, according to reaction 4, the re-produced CO_2 reacts with more carbon to produce more CO [12,18].

$$C + O_2 \rightarrow CO_2 \qquad (3)$$

$$CO_2 + C \rightarrow 2CO \qquad (4)$$

$$2CO \rightarrow CO_2 + C_{atom} \qquad (5)$$

Na_2CO_3 as an activator material is decomposed at elevated temperatures and produces CO_2 (reaction 6).

$$Na_2CO_3 \rightarrow Na_2O + CO_2 \qquad (6)$$

By producing CO_2 in the pack, the pressure of CO_2 increases. According to reaction 4, CO_2 reacts with carbon atoms present in the pack and produces CO. Fig. 4 shows the equilibrium diagram for reaction 5 at any temperature [1].

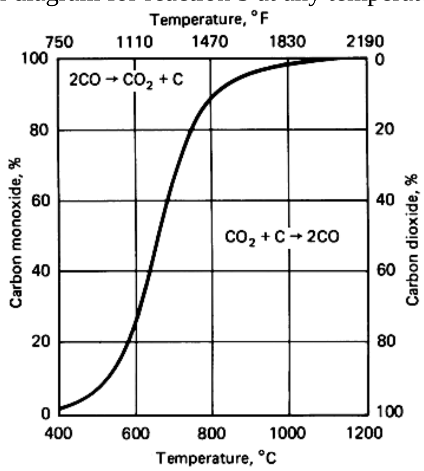

Fig. 4. Equilibrium pressure of CO and CO_2 for $2CO \rightarrow CO_2 + C_{atom}$ reaction [1]

According to Fig. 4, if the ratio of CO to CO_2 at a constant temperature is more than the equilibrium ratio of CO to CO_2, reaction 5 goes in the right direction, and the carburizing phenomenon happens. However, when the ratio of CO to CO_2 at a constant temperature is less than the equilibrium ratio of CO to CO_2, reaction 5 goes in the left direction, and the decarburizing phenomenon happens. When carbon diffuses into steel, the carbon content in the surface increases compared to the core of the steel, so that after quenching the steel in the oil, the hardness of the surface increases and the core remains flexible. According to Fig. 3 by increasing activator material up to 11.5 wt. %, ECD value was increased and then declined. In other words, up to 11.5 wt.% activator, the pressure ratio of CO to CO_2 in the carburizing box is more than the equilibrium ratio of CO to CO_2. In fact, up to this amount, the activator material decomposes continuously and according to reaction 6, the pressure of CO_2 in the carburizing pack increases continuously. The produced CO_2 gas reacts with the carbon atoms within the pack according to reaction 4 and CO gas is produced. CO causes carburizing phenomenon on exceeds this amount, decarburizing phenomenon happens and the case depth decreases. In this situation, more Na_2CO_3 is decomposed, and more CO_2 gas is produced. A small amount of CO_2 gas enters reaction 4 and produces CO, and a tremendous part of this gas causes oxidation in the box environment. As a result, CO_2 attracts atomic carbon from the surface of steel according to reaction 7.

$$CO_2 + C_{atom} \rightarrow 2CO \quad (7)$$

IV. CONCLUSIONS

Pack-carburization of 1.5920 cementation steel in the presence of Na_2CO_3 as an activator material was investigated using General Factorial Design of Experiment (GFDE). The conclusions drawn from the results can be summarized as follows:

(1) The optimum condition was achieved when $Na_2CO_3 \approx 11.5 wt.\%$ and in this range, time had not so important effect.
(2) The maximum ECD was obtained using 11.5 wt. % activator materials (Na_2CO_3) for the carburizing time of 12 hrs.
(3) At the constant time, by increasing the Na_2CO3 content from 5 to 11.5 wt. %, the Effective Case Depth (ECD) increases due to the carburizing phenomenon and afterwards decreases due to the decarburizing phenomenon.

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