

## Investigation on Chromizing of C45 Steel using Response Surface Methodology

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**Abstract:** The application of C45 steel in shafts studs, general fasteners and keys demand a hard and wear resistance surface. General heat treatment processes improve the core hardness resulting in brittleness of the components. In the present work, pack chromizing is done on C45 steel shafts to improve the surface (Vickers) hardness with a required diffusion depth. The core hardness is maintained at nominal value. The paper investigates the vitality of response surface methodology (RSM) in predicting the optimal combination of pack chromizing parameters to achieve the desired depth of diffusion and surface hardness. Experiments are conducted using Taguchi's  $L_{18}$  orthogonal array design and the optimal chromizing parameters are endorsed. The microstructure showing the diffusion depth obtained using optimal parameter setting is also studied.

**Keywords:** Chromizing, Taguchi, C45 steel, Response surface methodology, Vickers hardness, Optimization, Diffusion depth.

### 1 Introduction

Chromizing is the process of introducing chromium into a metal or a metal alloy. It is a surface hardening and thermo chemical heat treatment process performed by diffusing chrome atoms into the surface of the part to be treated at high temperatures. The process of chromizing can produce a hard, low-friction and wear-resistant surface compared to the other processes like boronizing and nitriding [1]. These thermochemical heat treatments are carried out in electrically heated furnace under controlled environmental conditions. A proper control can be established over the process by selecting and identifying the best level of chromizing parameters which can lead to the desired properties in the treated components. Generally in chromizing process, the surface hardness, finish and diffusion depth are observed as the required quality characteristics.

The process of pack chromizing is considered as a multi input and multiple response treatment and optimal combination of chromizing parameters can be identified by using methods like technique for order of preference by similarity to ideal solution (TOPSIS), grey relational analysis (GRA), principal component analysis (PCA), fuzzy logic and response surface methodology (RSM). Application of GRA and PCA is observed to yield the best operating condition. The combined approach is visualized to utilize the merits of both the techniques followed for optimization [2]. TOPSIS combined with the grey theory is also effective in hybrid format to sort the best set of inputs [3]. The grey Taguchi methods are very effective in single response optimization.

The RSM is a statistical method employed for modelling the quality characteristics which can be further optimized by using the desirability analysis [4, 5]. The generated response surfaces are used to study the

effects of different process parameters which can further be related to the mechanism of the process. The RSM technique is generally supplemented by the desirability analysis [6, 7, 8]. Generally central composite design (CCD) is used in experimentation and further analysis using RSM. However Taguchi's orthogonal array can also be employed for conducting the trials [9, 10, 11]. The desirability analysis is also found to yield good results in finding the optimal input conditions and the experimental strategy can also follow the Box–Behnken design [12]. However the results are arrived by experimenting with an excessive number of experimental trials. Hence Taguchi's orthogonal array is used for experimentation and are found to effective as well [13, 14].

From the literature, it is observed that proper selection of chromizing parameters can lead to better microhardness and typical applications of C45 steel can be improved by surface treatment using chromizing. Further little attention is noticed in optimization of chromizing parameters using RSM, especially when applied to C45 steel. Hence an attempt is made to use the technique of RSM for optimizing the chromizing parameters for C45 steel.

## 2 Experimentation

The material chosen for chromizing is C45 steel with a chemical composition as shown in Table 1. The C45 steel rods are subjected to thermo-chemical treatment in an electrically heated furnace rated at 24 KW and capable of generating a maximum temperature of 950°C using a nichrome heating element. The furnace retort is 500 mm in depth and 250 mm in diameter. The heat treatment enclosure is made of SS304L for sustaining heat and chemical inertness. The chrome ions are diffused into C45 steel specimens by pack chromizing method using a commercially available chromizing agent called WCT Cr-Cote. It basically comprises of a proprietary blend of chrome liberating agent, activator, diluter. The chromizing agent along with the specimen is packed in the enclosure with proper sealing.

**Table1** L<sub>18</sub> array displaying the experimental settings and obtained responses

C	Si	Mn	P	S	Fe
0.45	0.25	0.8	0.03	0.28	Remaining

The chromizing parameters included in the study are chromizing time, chromizing temperature and quenching temperature (Table 2). The parameters and their levels are selected based on preliminary chromizing trials and engineering judgement. Three levels are chosen for each parameter to perform pack chromizing. Taguchi's L<sub>18</sub>orthogonal array is used for conducting the chromizing trials.A central composite design is used for reducing the number of trials. After removing the enclosure with specimen, it is air cooled to room temperature, soaked in water and subjected to mild mechanical vibration to remove the residual chemical agents. The micro hardness (VHN) of the chromized specimen is observed at three places using a Vickers hardness tester by following the ASTM E384-2011 standard. A load of 100 g is applied using a diamond indenter for 15 s to measure the VHN. A section of the sample is prepared for observing the microstructure after grinding and subsequent polishing. The cylindrical specimens are treated with 2% nital etchant and the diffusion depth is measured at 200X (ASTM B487-2002 standard). An optical microscope inbuilt with a digital micrometer is employed for the same and the observed responses are listed in Table 3. The photograph of a few chromized specimen is shown in Figure 1.

**Table 2** Chromizing parameters and their levels

Notation	Process parameters	Levels Level 1		
		1	2	3
A	chromizing time (hr)	4	5	6
B	chromizing temperature (°C)	850	900	950
C	quenching temperature (°C)	750	800	850



**Figure 1** Photograph of a few chromized and treated samples

**Table 3**  $L_{18}$  array for experimentation and the obtained responses

Sl. No	Parameters			VHN		Diffusion depth		Core hardness	
	A	B	C	R1	R2	R1	R2	R1	R2
1	4	850	750	1308.52	1305.46	12.36	12.35	52	51
2	4	900	800	1346.28	1340.73	12.52	12.84	51	51
3	4	950	850	1389.34	1374.52	12.57	12.62	48	46
4	5	850	750	1405.63	1420.45	12.81	12.97	45	44
5	5	900	800	1455.33	1462.62	13.35	13.03	46	47
6	5	950	850	1576.42	1570.42	13.9	13.97	44	45
7	6	850	800	1528.63	1541.71	13.37	13.45	36	38
8	6	900	850	1557.74	1535.32	13.98	13.93	37	39
9	6	950	750	1573.91	1589.54	13.53	13.51	35	36
10	4	850	850	1423.11	1427.02	12.46	12.56	55	53
11	4	900	750	1412.48	1422.92	13.74	13.67	44	42
12	4	950	800	1433.62	1425.27	13.42	13.28	46	44
13	5	850	800	1450.11	1442.96	12.54	12.72	49	48
14	5	900	850	1469.34	1479.54	12.63	12.86	47	46
15	5	950	750	1483.53	1488.88	13.78	13.45	43	42
16	6	850	850	1537.93	1532.76	12.87	12.68	38	39
17	6	900	750	1535.53	1537.92	13.36	13.54	41	41
18	6	950	800	1555.83	1578.96	13.32	13.67	38	39

### 3 Modelling and Optimal Level Selection using RSM and desirability Analysis

After performing the process of pack chromizing on C45 steel, the experimental results are analysed using RSM. Models are formed for the various responses and desirability analysis is employed to sort out the optimal parameter combination for chromizing. The various steps involved are shown below.

**Step 1:** Form mathematical models (second order polynomial equations) for the responses.

**Step 2:** Perform the analysis of variance (ANOVA) for the responses to supplement RSM [7].

**Step 3:** Plot the response surface graphs to study the parameter effects [4].

**Step 4:** Find the optimal level using desirability analysis [5, 6].

**Step 5:** Conduct the validation experiment.

## 4 Results and Discussions

### 4.1. Quadratic Models

The mathematical modelling for VHN (Eq. 1), diffusion depth (Eq. 2) and core hardness (Eq. 3) are performed using the Design Expert software to study the effects of parameters within the experimental domain. The model can be used to observe the interaction and individual effects of the chromizing parameters. The model coefficients are evaluated using the software and the ANOVA results are listed in Tables (4-6). The predicted and adjusted R-squared values are observed to agree well with each other and the higher values of adequate precision is also observed for these models. This proves the fitness, adequacy and statistical significance of the generated models.

**Table 4 ANOVA table for VHN**

Source	SS	Dof	MSS	F- Value	p-value	Remarks
Model	2.00E05	7	28496.23	30.02	< 0.0001	significant
A-Chromizing time	1.36E05	1	1.36E+05	143.27	< 0.0001	
B-Chromizing	21357.68	1	21357.68	22.5	< 0.0001	
C-Quenching temperature	6295	1	6295	6.63	0.0156	
BC	3554.51	1	3554.51	3.74	0.0631	
A <sup>2</sup>	1135.68	1	1135.68	1.2	0.2834	
B <sup>2</sup>	889.22	1	889.22	0.94	0.3414	
C <sup>2</sup>	761.35	1	761.35	0.8	0.3781	
Residual	26577.82	28	949.21			
Cor Total	2.26E+05	35				

**Table 5 ANOVA table for diffusion depth**

Source	SS	Dof	MSS	F- Value	p-value	Remarks
Model	5.7	7	0.81	6.44	0.0001	significant
A-Chromizing time	1.94	1	1.94	15.32	0.0005	
B-Chromizing temperature	3.13	1	3.13	24.73	< 0.0001	
C-Quenching temperature	0.2	1	0.2	1.62	0.214	
AB	0.033	1	0.033	0.26	0.6145	
AC	0.59	1	0.59	4.68	0.0393	
B <sup>2</sup>	0.16	1	0.16	1.29	0.265	
C <sup>2</sup>	0.025	1	0.025	0.2	0.6583	
Residual	3.54	28	0.13			
Cor Total	9.25	35				

**Table 6 ANOVA table for core hardness**

Source	SS	Dof	MSS	F- Value	p-value	Remarks
Model	889.56	8	111.19	35.6	< 0.0001	significant
A-Chromizing time	661.5	1	661.5	211.78	< 0.0001	
B-Chromizing	96.73	1	96.73	30.97	< 0.0001	
C-Quenching temperature	40.28	1	40.28	12.9	0.0013	
AB	52.6	1	52.6	16.84	0.0003	
AC	29.17	1	29.17	9.34	0.005	
A <sup>2</sup>	37.56	1	37.56	12.02	0.0018	
B <sup>2</sup>	4.71	1	4.71	1.51	0.23	
C <sup>2</sup>	8.21	1	8.21	2.63	0.1166	
Residual	84.33	27	3.12			
Cor Total	973.89	35				

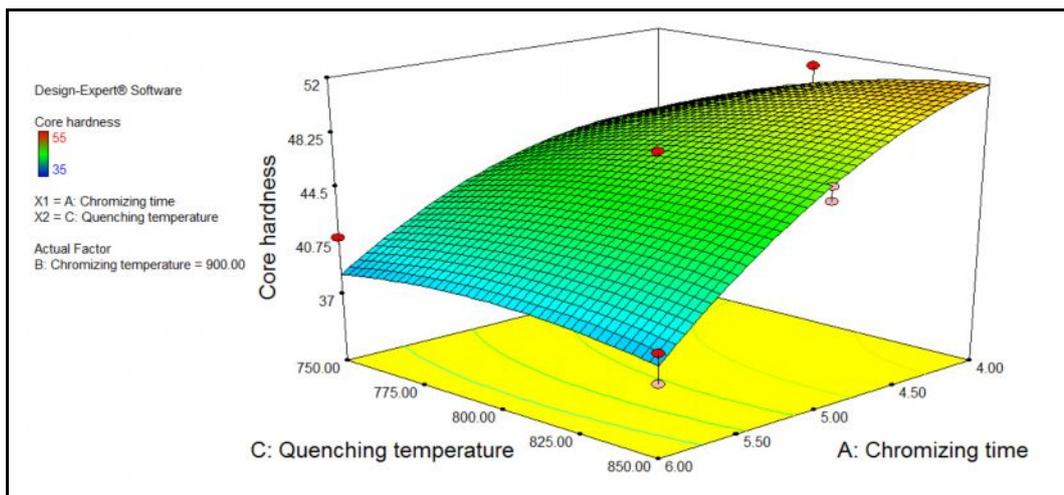
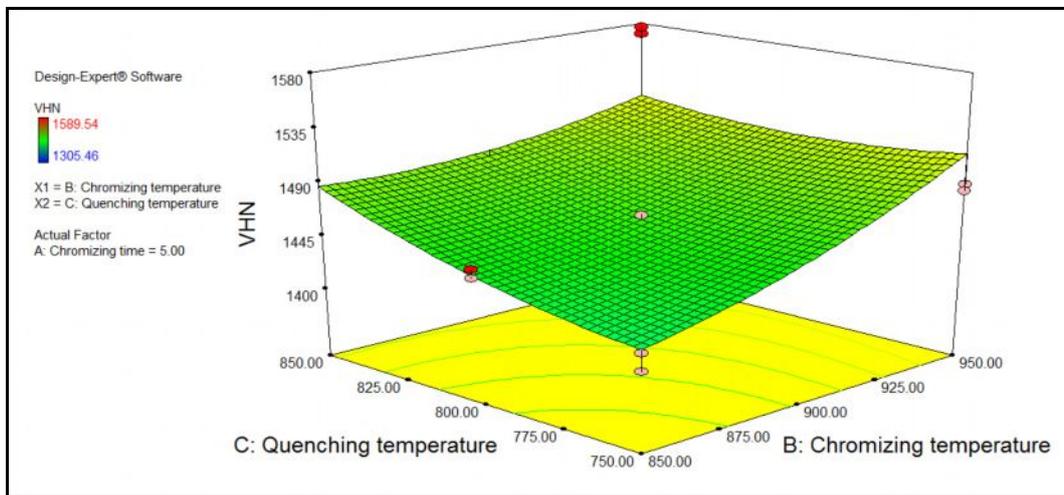
$$VHN = +1293.59417 + 200.46036 * A - 1.89538 * B - 0.18330 * C - 6.37362 \times 10^{-3} * B * C - 12.12560 * A^2 + 4.21717 \times 10^{-3} * B^2 + 3.90217 \times 10^{-3} * C^2 \tag{1}$$

$$\text{Diffusion depth} = -13.80383 - 2.15802 * A + 0.11732 * B - 0.059335 * C - 9.78125 \times 10^{-4} * A * B + 4.15313 \times 10^{-3} * A * C - 5.82344 \times 10^{-5} * B^2 + 2.28906 \times 10^{-5} * C^2 \tag{2}$$

$$\text{Core hardness} = -422.52083 + 4.50000 * A + 0.32437 * B + 0.83312 * C + 0.039167 * A * B - 0.029167 * A * C - 2.16667 * A^2 - 3.12500 \times 10^{-4} * B^2 - 4.12500 \times 10^{-4} * C^2 \tag{3}$$

**4.2 Response surface graphs**

The response surface (3D) graphs are developed by using Design Expert software. These plots can be used to estimate and study the effects of various chromizing parameters on the various responses observed via experimentations. It is observed from these plots, that a higher level of chromizing time and temperature can improve the diffusion depth and microhardness of the chromized surface. Hence better surface properties in the parent material. However the core hardness is not affected significantly. An increase in tempering temperature is found to improve the core hardness by a reasonable extent. However its influence on diffusion depth and microhardness of the chromized surface is found to be very less.



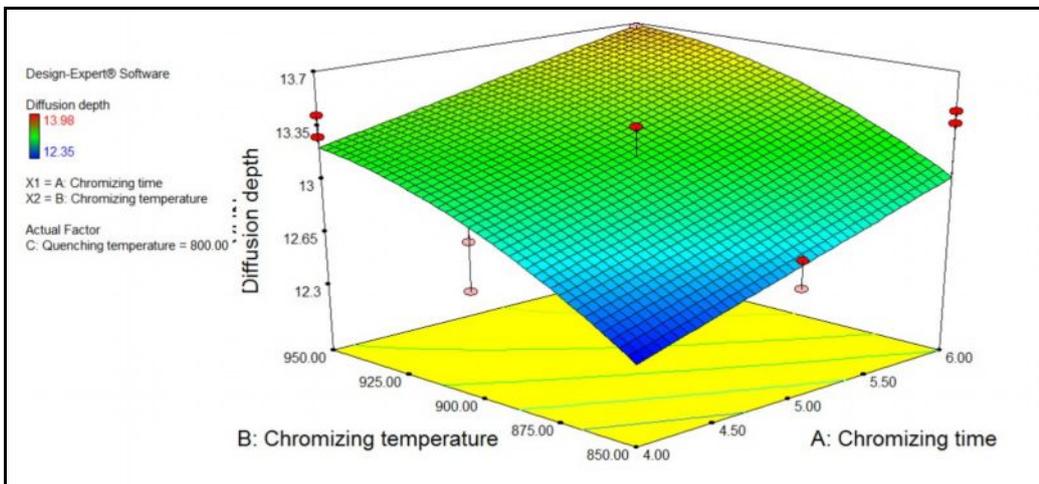
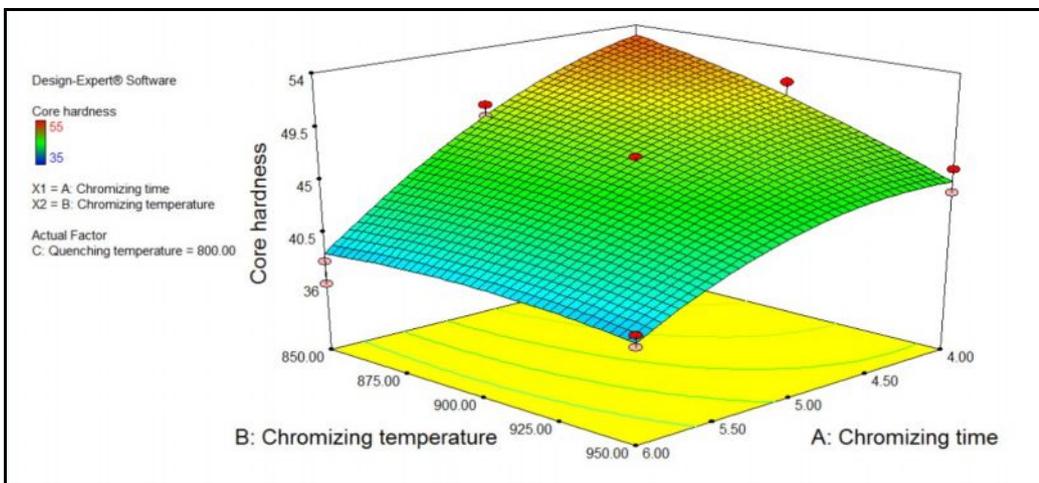
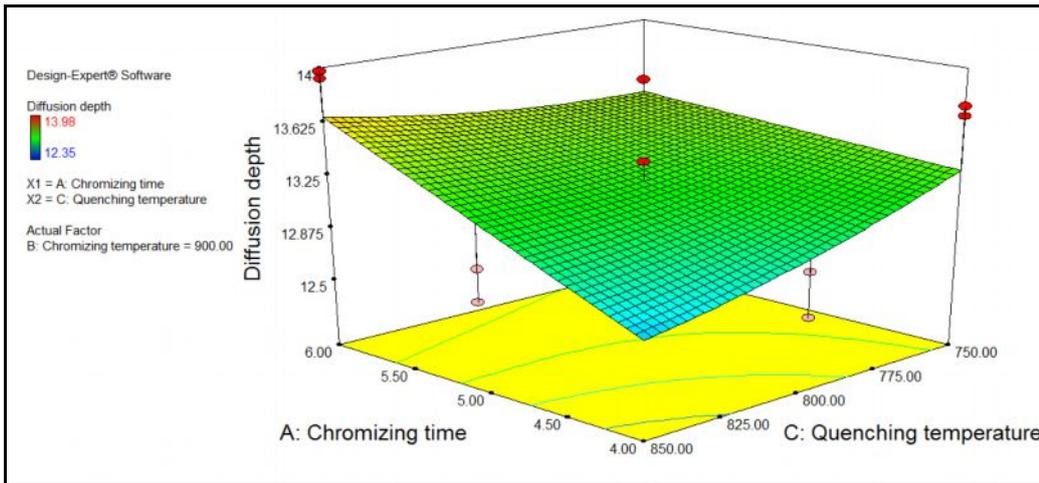


Figure 2 Response graphs

### 4.3 Ramp graph

The optimal parameters for chromizing the C45 steel are shown in Figure3. The levels are indicated by a dot on each ramp according to which the optimal level is chosen. The height of the dot indicates the magnitude of desirability, whose value lie between zero and one, based on the closeness of the responses with the target. The Figures (4-6) indicate a reasonable degree of proximity between the predicted and actual values of different responses. The distributed points and their vicinity towards the centre line proves the proximity.

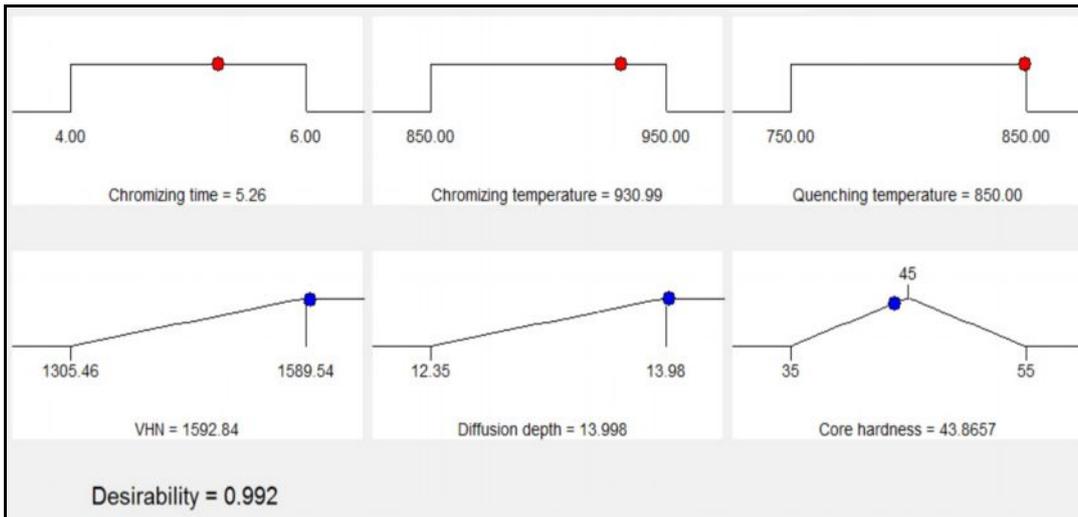


Figure 3 Ramp graph

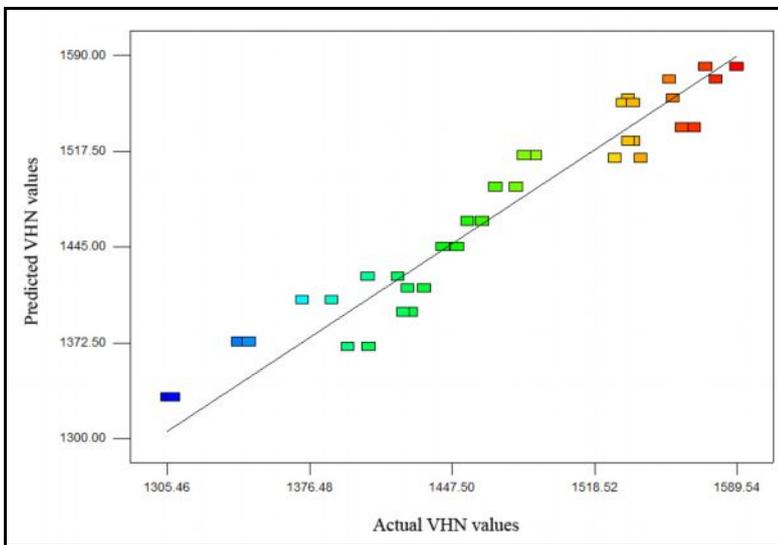


Figure 4 Plot of actual and predicted values of VHN

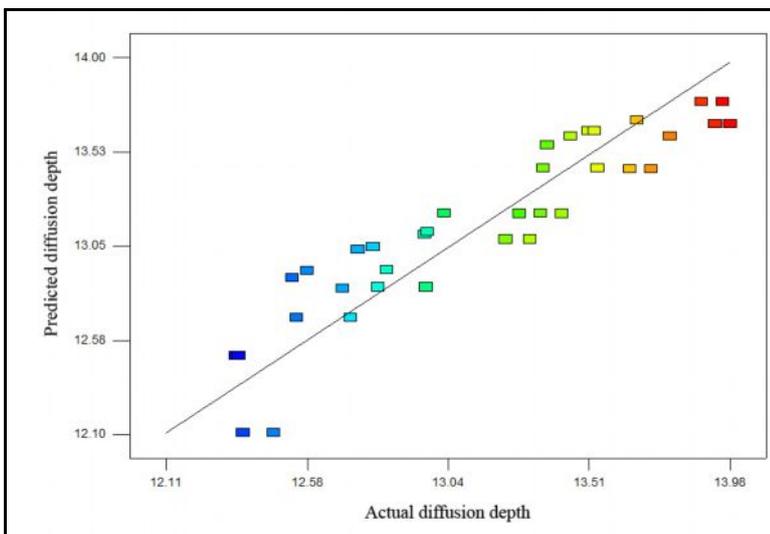


Figure 5 Plot of actual and predicted values of diffusion depth

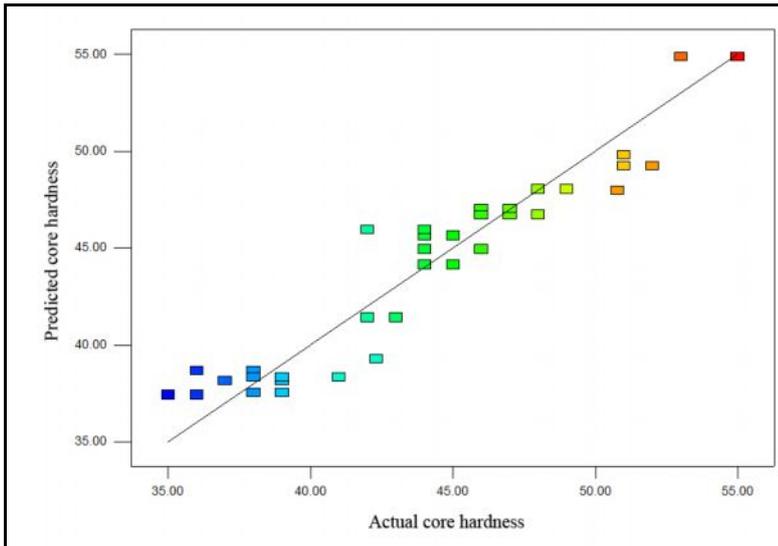


Figure 6 Plot of actual and predicted values of VHN

4.4. Optimal level selection from desirability analysis

The *larger-the-better* desirability function is used for the desirability analysis, performed by using the design expert software. The optimal levels are identified from Table 7. The optimal level is selected as A<sub>2</sub>B<sub>3</sub>C<sub>3</sub>. After obtaining the optimal chromizing parameter setting, a confirmatory experiment is conducted to prove its worth and validate the analysis. A good improvement in responses is observed which ensures the suitability of application of RSM in various machining processes. The microstructure observed with the optimal chromizing parameter setting is shown in Figure 7.

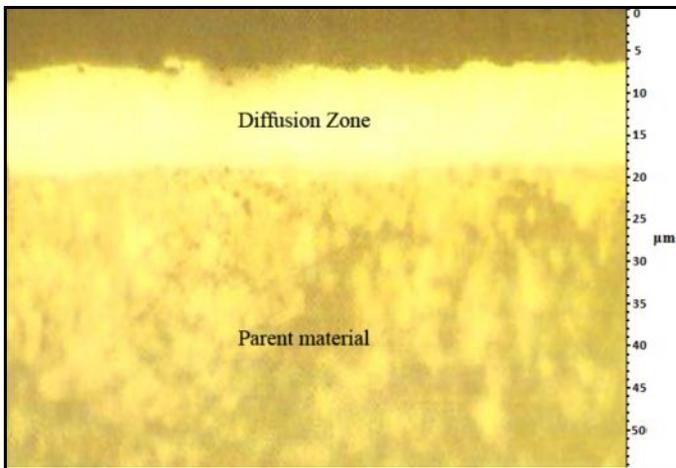


Figure 7Microstructure of the specimen chromized under optimal condition.

Table 7Desirability analysis and confirmatory trial

Notation	Parameters	Optimal level	Level 1	Level 2
A	Chromizing time	5.26	4	6
B	Chromizing temperature	930.98	850	950
C	Quenching temperature	850	750	850
Responses observed at optimal chromizing parameters				
VHN	1692.84			
Diffusion depth	14.99			
Core hardness	43.86			

## 5 Conclusion

The study discloses an informative report on pack chromizing of C45 steel widely used in shafts studs, general fasteners and keys. The application of RSM is validated and the following conclusions are revealed.

- The methodology of RSM is used to generate models for VHN, diffusion depth and core hardness in terms of various chromizing parameters.
- The generated models are observed to be fit, significant and adequate in representing the conditions for chromizing C45 steel.
- The usage of Taguchi's orthogonal array has permitted lesser experimental trials when compared to the CCD method employed with RSM.
- The research findings will offer the require guidelines and database for pack chromizing of C45 steel.

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