Investigation in Gas Carburizing of AISI 4140, EN36, and 16MnCr5 Steels Using the Grey Incidence-Based Taguchi (GIBT) Method

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Abstract

Gas carburizing is an effective surface treatment process for improving the hardness and wear resistance of different classes of steels. This study reports an application of grey-incidence based Taguchi (GIBT) method in gas carburizing of case-hardening steels like AISI 4140, EN36, and 16MnCr5 which are widely employed in precision levers, transmission shafts, and pinions. Carburizing trials are performed using Taguchi’s L9 orthogonal array by varying the design parameters like carburizing temperature, soaking time, and tempering temperature. Surface hardness (SH), diffusion depth (DD), and wear loss (WL) are studied as process responses at the completion of various carburizing trials with replications. Optimal design variables are identified using grey incidence grade as a performance index in the GIBT method. The contribution of individual parameters is also studied using the analysis of variance (ANOVA). Microscopic examination and SEM images of the treated surface are also studied after validating the method of GIBT.

1. Introduction

Surface hardening is an important process to improve wear resistance and hardness in machine parts like shafts, cam rollers, strips, pinions, etc. Gears used in automobiles and power mills encounter high specific loads due to greater torque requirements. Improving the life of such components including an offer of lifetime warranty was possible by surface treatments like carburizing. Gas carburizing is a case-hardening process in which carbon is diffused into the workpiece surface by heat treatment in an atmosphere of carbon carrying gas. Normally, methane or propane gas is used to create an atmosphere with carbon potential along with a neutral gas. The part is finally quenched and tempered to complete the sequence. Pack carburizing consumes time and hence is limited in applications. Gas carburization is found to be the more effective one than both vacuum and plasma carburizing. The process does not harden the steel directly; however, it improves the carbon content to a sufficient level beneath the surface for subsequent quenching and tempering [1]. Soaking time and carburizing temperature play a vigorous part in diffusion of carbon atoms and diffusion depth depend on these parameters. A controlled gas carburization could produce a well-defined carbon profile with an endothermic gas like carbon monoxide [2].

In most cases, the temperature was not increased beyond 980°C though faster carbon diffusion was possible at higher temperatures, saving the process time as well. Necessary care was essential to select an appropriate furnace and reduce the grain coarsening in parent steel. The gas carburizing...
parameters like holding time, soaking temperature, quench temperature, etc., influence the quality characteristics of the process. In a few cases, the carburized components were annealed to improve their machining characteristics, particularly during the final finishing operations [3]. AISI 1040 steels could be carburized in a gaseous environment to improve the wear resistance characteristics and the wear properties were observed to vary directly with the soaking temperature [4]. SAE-40 oil could be used as a quenching medium at 100°C for 5 min to prevent the distortion of thermally treated specimen and prepare it for tempering [5]. The case-hardening steels like AISI 4140, EN36, and 16MnCr5 are used in precision and high tolerance applications which demand a harder surface with good core toughness. Plasma carburizing of aluminium alloy at relatively lower temperatures could improve the corrosion resistance of surface-treated parts [6]. Proper quality control was possible by selecting parameters like carburizing temperature, soaking time, and tempering temperature at optimal levels.

Optimization of multiple responses in a manufacturing process is relatively complex compared to single response optimization. Optimization is an effective offline quality managing tool for identifying the good combination of parameters. Multiresponse optimization can be solved by methods like TOPSIS, grey relational analysis (GRA), response surface method, back propagation networks, and principal component analysis [7]. Optimization of surface roughness was possible with the Taguchi method and genetic algorithm. The experimental trials were designed based on orthogonal arrays; however, the selection of parameters and their levels requires a good amount of skills [8]. Orthogonal array and variance analysis (ANOVA) was used to improve the machining responses, while the parameter effects were studied using the Taguchi technique and three dimensional plots [9, 10]. The Taguchi method was effectively used with orthogonal arrays to find the optimal conditions. In such instances, the signal-to-noise ratio was used to arrive at the optimal design variables [11, 12]. The Taguchi method follows the basic statistical design approach for improving the quality in various manufacturing practices. The robust method uses minimal experimental trials to study the design variables hence saving time as well as cost. The trials runs were chosen specifically to exclude the variations in measured responses due to noise [13]. GRA was used to find the optimal conditions along with the Taguchi method in different manufacturing processes effectively. Grey relational grade was used as an index in such instances along with the signal-to-noise ratio [14]. The quality characteristics were generally subjected to normalization during analysis. The combination of the Taguchi method and grey theory was highly effective in different manufacturing practices [15, 16]. GRA could handle the uncertainties better by forming the grey relational grade which could be used as a single representative for the multiple responses [17]. GRA was used in its original form or in combination with other methods like TOPSIS, genetic algorithm, fuzzy, and back propagation networks in various instances of decision making and in different production processes to arrive at the optimal parameters, thus ensuring the possibilities of hybridization of techniques [18–20].

From the existing literature, it was understood that case-hardening steels like AISI 4140, EN36, and 16MnCr5 require a high surface hardness with good core toughness in most of their applications which demand a high technical maturity. Parameter design in carburizing of case-hardening steels was observed to be limited in literature. Hence, a novel attempt was made to apply grey incidence-based Taguchi (GIBT) method on gas carburizing of AISI 4140, EN36, and 16MnCr5 steels. The predicted optimal parameters and developed carburizing guidelines will assist the industries handling case-hardening steels.

2. Experimental Design of Surface Treatment Trials

The creep resisting feature of case-hardening steels (AISI 4140, EN36, and 16MnCr5) is good. Further, these class of steels have the capability to retain their mechanical properties at higher temperatures. The substrate materials for surface heat treatment (carburizing) were procured as circular rods of diameter 18 mm which were machined to a diameter of 15 mm. Finally, the specimen to be treated in the furnace was cleaned with acetone to eliminate dirt, grease, and oil. The gas carburizing furnace (Figure 1) employing a Nikrothal® heating element and capable of attaining a maximum temperature of 1200°C was used for experimentation. The furnace, rated at 30 kWh, was made of creep resisting retorts and includes a thermocouple of K-type. The prepared sample was kept inside the tightly sealed furnace and required temperature is generated. The specimen was allowed to soak in an atmosphere of a carbon-carrying gas (carbon monoxide). The same atmosphere was used for tempering after reducing the carbon concentration. Finally, the surface-treated specimen was subjected to testing for observing the microhardness, depth of diffusion, and loss due to wear.

2.1. Carburizing Parameters and Responses. The carburizing parameters, which play an important role in determining the responses, were chosen from literature [2–4]. The following parameters were varied for surface treatment: carburizing temperature, soaking time, and tempering temperature. Pilot carburizing trials were conducted with industrial expertise to find the levels of parameters yielding better surface characteristics. After carburizing, all samples are soaked in SAE oil at 100°C and tempered for an hour to complete the treatment sequence. The parameters for carburizing are shown in Table 1. Surface treatment trials were carried out using orthogonal array (L9) for two replications which was a proven tool for studying the quality characteristics of a process [13, 14]. Optimization was basically an offline technique for control of quality characteristics. Surface hardness (SH), diffusion depth (DD), and wear loss (WL) were observed after each trial performed in a random sequence [7].
The Vickers microhardness tester (loading capability: 0.01 kg to 2 kg) was used to measure microhardness following ASTM B487-2002 standard. The microhardness values were studied for a load of 100 g for 10 s with an indenter made of diamond. The observations were made at five different spots in the treated surface and the mean values were tabulated for analysis. A pin-on-disc wear tester was used to find wear loss at 10 N load using alumina. Wear resistance was found using Saini make, SSI-114 pin on disc (PoD) tester for a time period of 600 s at a sliding velocity of 1.5 m/s. The wear loss was measured using a digital weighing balance (model: P7, Kerro make) with an inbuilt timer. The specimen polished in a silica suspension was subjected to buffing and etching for 40 s with Nital solution, before observing the same under a microscope to find the diffusion depth. The sample specimen subjected to surface treatment is shown in Figure 2, while the responses are shown in Table 2.

The maximum value (603 HV) and the least value (570 HV) of surface microhardness were observed during trial 7 and trial 6, respectively. Trial 7 corresponds to input condition: material substrate-AISI 4140 steel, carburizing temperature-860°C, soaking time-6 h, and tempering temperature-260°C. Diffusion depth on the higher side was an indication of an improved surface in terms of resistance to wear and corrosion. The diffused carbon atoms have the tendency to form carbides on the steel surface which along with the conversion of a portion of austenite to martensite could produce superior grade surfaces [3]. The observed wear loss was the highest (0.0238 gm) in trial 5 and the lowest (0.0193 gm) in trial 8. Trial 8 corresponds to input condition: material substrate-AISI 4140 steel, carburizing temperature-940°C, soaking time-5 h, and tempering temperature-220°C. A higher value of diffusion depth and surface hardness was desired, while the wear loss was preferred to be on the minimal side. The values of obtained responses (two replications) are shown in Table 2.

2.2. Microstructural Examination. The microstructure of a material could play a vital role in determining the properties like strength, toughness, wear resistance, and hardness [9, 13]. These properties could determine the performance of material substrates in typical applications; hence, a microstructural study becomes essential. The microstructure of AISI 4140, EN36, and 16MnCr5 steels was analysed after carburizing at prescribed conditions. The microstructural examination was done to find the case depth and defects, if any, in the specimen as per ASTM E3 standard. The specimen of three different material substrates were moulded in Bakelite (Figure 3). The surface was subjected to buffing and treated with 3% Nital solution which acts as an etchant. The case and core microstructure for all the case-hardening steels are presented in Figure 4. The darkest region represents the mould, while the case depth indicating the diffusion of carbon atoms was seen in treated surfaces. The core of the material substrates remains unaffected and free of defects. The darker region in the microstructure indicates presence of carbides which improve the surface hardness of treated specimen and hence its tribological properties.
3. Grey Incidence-Based Taguchi (GIBT) Method

The multiresponse optimization is an offline method for quality control, predicting more practical solutions [7]. The grey theory deals with sorting out the uncertainty in a specific system of incomplete data. The grey incidence is a normalization-based technique to find out the best set of input conditions from a discrete data. The strength of grey incidence analysis lies in system modeling with lesser information [13]. Prepossessing of data is essential to decrease the noise and randomness in GRA, which follows a unique theory to arrive at the decision. The Taguchi technique involving the ‘signal-to-noise’ ratio (S/N ratio) was used to compare outputs and grey theory analysis was used to find the relation among elements based on the similarity of trends [17]. The grey incidence-based Taguchi (GIBT) method comprises of the following steps.

3.1. Grey Incidence Generation. The S/N ratio (\( \eta \)) for each quality characteristic was found out using (1) and (2) for ‘smaller-the-better’ and ‘larger-the-better’ type responses, respectively [7]. The smaller-the-better analysis was done for wear loss, while the remaining responses (diffusion depth and surface hardness) were larger-the-better conformities. Each carburizing trial was performed twice (replication) for better accuracy. Further normalization was important to decrease the variability among the S/N ratio. It was a min-max scaling procedure to re-scale the observed output (\( y_{ij} \)) values in the interval of 0 to 1.

\[
\frac{S}{N\text{Ratio}}(\eta) = -10 \log_{10} \left( \frac{1}{r} \sum_{i=1}^{r} y_{ij}^2 \right), \tag{1}
\]

\[
\frac{S}{N\text{Ratio}}(\eta) = -10 \log_{10} \left( \frac{1}{r} \sum_{i=1}^{r} \frac{1}{y_{ij}} \right). \tag{2}
\]

Normalization was done using (3), before arriving at the grey incidence coefficient (GIC) and grey incidence grade (GIG) values using Equation (4) and Equation (5), respectively. GIC relates the observed and actual results, while GIG values act as a single representative. The GIG values are shown in Table 3. The calculation of GIG values efficiently reduces the different process responses to a single entity. Hence, the multiresponse optimization scenario in carburizing of case-hardening steels (AISI 4140, EN36, and 16MnCr5 steel) was effectively condensed to optimization of a single entity (GIG). The value of distinguishing coefficient was taken as 0.33 to assign an equal weight for all the three process outputs.

\[
z_{ij} = \eta_{ij} - \min \left( \eta_{ij}, i = 1, 2, \ldots, n \right) = \min \left( \eta_{ij}, i = 1, 2, \ldots, n \right) - \eta_{ij}, \tag{3}
\]

\[
y_{ij}^* = \Delta \min \frac{+\xi \Delta \max}{\Delta_o(i) + \xi \Delta \max}, \tag{4}
\]

\[
GIG_i = \sum_{i=1}^{n} (y_{ij}). \tag{5}
\]

where \( r \)-number of replications, \( m \)-number of observations, \( \Delta \min = \min \| z_{o}(i) - z_{ij}(i) \| \) for all \( i \) and \( jej \), is the least value of \( z_{ij}(i) \); \( \Delta \max = \max \| z_{o}(i) - z_{ij}(i) \| \) for all \( i \) and \( jej \) is the highest value of \( z_{ij}(i) \), and ‘\( \zeta \)’ is the necessary distinct coefficient.

The GIG values for each trial is shown graphically in Figure 5. These GIG values serving as the rating index do not follow any pattern as seen from the curve. The largest GIG value (0.8590) was observed for trial number 7 (material substrate-AISI 4140 steel, carburizing temperature-860°C, soaking time-6 h, and tempering temperature-250°C), displaying the apt levels of carburizing inputs within experimental field for improved responses (surface hardness, diffusion depth, and wear loss) in terms of GIG. The smallest
Figure 4: Microscopic examination of AISI 4140, EN36, and 16MnCr5 steels.

Table 3: Quality index (GIG values).

| Trial | S/N ratio | Normalized S/N ratio | Grey incidence coefficient | GIG |
|-------|-----------|----------------------|----------------------------|-----|
|       | SH        | DD       | WL    | SH  | DD  | WL    | SH  | DD  | WL    |       |
| 1     | 55.276    | 35.265   | 33.271| 0.307| 0.753| 0.450 | 0.419| 0.669| 0.476 | 0.5216|
| 2     | 55.321    | 35.848   | 33.146| 0.403| 0.862| 0.379 | 0.456| 0.784| 0.446 | 0.5619|
| 3     | 55.186    | 35.559   | 32.770| 0.114| 0.808| 0.165 | 0.361| 0.722| 0.374 | 0.4859|
| 4     | 55.343    | 35.703   | 34.172| 0.451| 0.835| 0.965 | 0.477| 0.752| 0.935 | 0.7210|
| 5     | 55.283    | 36.585   | 32.482| 0.323| 1.000| 0.000 | 0.425| 1.000| 0.333 | 0.5861|
| 6     | 55.133    | 35.833   | 33.127| 0.000| 0.859| 0.368 | 0.333| 0.780| 0.442 | 0.5185|
| 7     | 55.599    | 35.489   | 34.100| 1.000| 0.795| 0.924 | 1.000| 0.709| 0.868 | 0.8590|
| 8     | 55.336    | 31.243   | 34.233| 0.435| 0.000| 1.000 | 0.469| 0.333| 1.000 | 0.6009|
| 9     | 55.417    | 35.984   | 33.566| 0.610| 0.887| 0.619 | 0.562| 0.816| 0.567 | 0.6484|
value of index (0.4859) was obtained for trial number 3 (material substrate-EN36, carburizing temperature-940°C, soaking time-6h, and tempering temperature-280°C), indicating relatively reduced responses.

3.2. Effects of Parameters on Responses. The effects of various surface treatment parameters are studied from plots shown in Figures 6(a)–6(d) using normalized S/N ratio. It was observed that AISI 4140 subjected to carburizing revealed a moderate diffusion depth, lesser wear loss, and an improved surface hardness as evident from Figure 6(a). Hence, among the case-hardening steels subjected to carburizing, AISI 4140 had shown better responses within the range of experimentation. A lesser level of carburizing temperature allows for an optimal diffusion of carbon atoms on surface as observed from Figure 6(b). An elevated temperature enhances carbon potential inside in furnace, approving diffusion of more carbon atoms [2]. However, an enhanced diffusion could also create a soft carbon deposit which was undesirable as it decreases surface hardness. Hence, a lower level of temperature could produce better characteristics. A moderate level of soaking time (Figure 6(c)) prevents formation of soft spots over the surface, creating lesser cleaning problems and an improved surface property. It was also observed that increased soaking time beyond 5 h does not create noteworthy changes or improvements in quality characteristics, as the increased carbon deposit was observed to degrade the microhardness of the treated surface. Further, the diffusion depth tends to decrease because of carbon saturation on the surface beyond 5 h of soaking time.

Diffusion of carbon beneath the surface of treated components results in formation of iron carbides [1, 3]. On a microscopic scale, these iron carbides could generate necessary amount of resistance to dislocation movement or slip of crystals. This could be the prime reason for an improved surface hardness. Further, carbon-rich specimen skin with a few portion of austenite converted to harder martensite could add a vital edge to improvement in surface properties [1, 3]. Generally, tempering at higher temperature could decrease the hardness mildly and it could be accompanied by a significant wear loss [4]. A moderate level of tempering temperature could also improve the surface properties as seen from Figure 6(d). The usage of L9 orthogonal array limits the study to observing the effects of individual parameters in carburizing and not their interaction effects. The optimal carburizing parameters for AISI 4140 steel were identified using min-max criteria as follows: carburizing temperature-860°C, soaking time-5 hours, and tempering temperature-250°C.

3.3. Analysis of Variance (ANOVA). Generation of errors in testing of hypothesis due to inflation of significance level could be resolved by using the analysis of variance (ANOVA). This would permit the comparison of means and variances. The variance analysis could identify the contribution of carburizing parameters using the F-values. ANOVA was performed on the GIG values which represent the individual responses. The values of sum of squares (SS) and mean sum of squares (MS) are shown in Table 4. They form the vital measures of data variability while the statistical significance of the test was indicated by the F value [7]. The process of carburizing had produced better surface properties with AISI 4140 substrate than other studied steels. Carburizing temperature was identified to affect the responses significantly with a contribution of 33.74%, compared to soaking time whose contribution was 16.95%. The statistically insignificant parameter (tempering time) produced a lesser contribution on GIG, and hence, it was pooled into error.

4. Validation of the Optimal Parameter Level

Parameter design was a robust method of obtaining the better design variables. It could effectively reduce the variations in observed quality responses due to noise, hence providing the scope for developing high quality systems [7]. The grey incidence-based Taguchi (GIBT) method was used to identify the optimal parameter setting using max-min criteria as follows: material substrate-AISI 4140 steel, carburizing temperature-860°C, soaking time-5 h, and tempering temperature-250°C. The validation of identified optimal setting within the experimental range was desired to ensure the effectiveness of GIBT method in carburizing of case-hardening steels. The results of validation experiments are shown in Table 5. The responses (surface hardness, diffusion depth, and wear loss) obtained with predicted optimal settings were compared with those produced with initial setting of parameters (AISI 4140 steel, carburizing temperature-860°C, soaking time-6 h, and tempering temperature-250°C). This initial setting corresponds to the combination of carburizing parameters within experimental range (trial 7) which had produced the largest value of GIG (0.8590). Considerable improvement in quality characteristics was observed with setting identified by GIBT approach. A surface hardness improvement of 17 HV was observed along with an improvement in diffusion depth by 12 μm. The percentage improvement in wear loss was found out as 7.61.
Figure 6: Parameter effects on quality characteristics. (a) Material substrate, (b) carburizing temperature, (c) soaking time, and (d) tempering temperature.

Table 4: Pooled ANOVA on GIG.

| Parameter                  | SS     | DOF | MS     | F-ratio | % Contribution |
|----------------------------|--------|-----|--------|---------|----------------|
| s-Substrate material       | 0.0485 | 2   | 0.0242 | 8.1356  | 43.91          |
| Bc Carburizing temperature | 0.0372 | 2   | 0.0186 | 6.2519  | 33.74          |
| s-Soaking time             | 0.0187 | 2   | 0.0094 | 3.1403  | 16.95          |
| Error                      | 0.0060 | 2   | 0.0030 |         | 5.40           |
| Total                      | 0.1104 | 8   |        |         | 100            |
The scanning electron microscopy (SEM) images exhibiting diffusion depth (AISI 4140 substrate) for initial and optimal factor setting are revealed in Figures 7(a) and 7(b). Diffusion depth observed was larger in case of carburizing with predicted optimal parameters (material substrate-AISI 4140 steel, carburizing temperature-860°C, soaking time-5 h, and tempering temperature-250°C) compared to one obtained with trial 7 setting with better value of GIG (material substrate-AISI 4140 steel, carburizing temperature-860°C, soaking time-6 h, and tempering temperature-250°C). The darkest region corresponds to the mould material (Bakelite), while a relatively darker layer of diffused carbon prompts the formation of iron carbides (Fe₃C), improving the surface hardness [1, 3]. However, the toughness of core observed at the bottom of image was maintained. Hence, the metallurgical surface modification brought by the diffusion of carbon atoms into the AISI 4140 substrate had produced a blockage to microscopic slip, increasing the hardness and resistance to wear as well [2, 6].

### 5. Conclusions

The investigation discloses the maiden application of grey incidence-based Taguchi (GIBT) method in gas carburizing of case-hardening steels (AISI 4140, EN36, and 16MnCr5) used in precision levers, transmission shafts, and pinions. The merits of the grey incidence theory and Taguchi method were integrated in the approach of GIBT to predict the optimal parameter combination resulting in an improved wear loss, diffusion depth, and hardness of treated surface.

Carburizing trials were performed using Taguchi’s L9 orthogonal array and following interpretations were drawn.

(i) Generally, an improved surface was obtained by gas carburizing of case-hardening steels (AISI 4140, EN36, and 16MnCr5), and the GIG value was used as performance index to represent the quality characteristics (surface hardness, diffusion depth, and wear loss) as a single entity in the GIBT method.

(ii) Carburizing temperature was identified to affect the responses significantly with a contribution of 33.74%, compared to soaking time whose contribution in influencing the surface characteristics was 16.95%.

(iii) The grey incidence-based Taguchi (GIBT) method was used effectively to find the optimal condition for carburizing of case-hardening steel: material substrate-AISI 4140 steel, carburizing temperature-860°C, soaking time-5 h, and tempering temperature-250°C.

(iv) Considerable improvement in quality characteristics were observed with setting identified by GIBT approach. A surface hardness improvement of 17 HV was observed along with an improvement in diffusion depth by 12 µm. The wear loss improvement was found out as 7.61%, hence validating the approach in experimental domain.

(v) A relatively darker layer of diffused carbon prompts the formation of iron carbides (Fe₃C), improving the surface hardness. Greater diffusion depth was
observed on surface of AISI 4140 substrate than EN36 and 16MnCr5 steels.

The study findings can offer significant guidelines for surface treatment of case-hardening steels like AISI 4140, EN36, and 16MnCr5 by gas carburizing. This will contribute to enhance their applications in pinions, transmission shafts, levers, and strips, opening the possibility of adopting the GIBT approach in other surface treatment processes as well. Further studies to analyse the interaction effects of carburizing parameters could add value to the subject in future.

Abbreviations
\[ \eta: \text{Signal-to-noise ratio} \]
\[ y_i: \text{Observed response} \]
\[ Z_i: \text{Normalized value of the S/N ratio} \]
\[ \xi: \text{Distinct coefficient} \]
\[ Y: \text{Grey incidence co-efficient} \]

Data Availability

The data used to support the findings of this study are included in the article. Should further data or information be required, these are available from the corresponding author upon request.

Disclosure

It was performed as a part of the Employment Hawassa University, Ethiopia.

Conflicts of Interest

The authors declare that there are no conflicts of interest regarding the publication of this paper.

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