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EFFECT OF LOW-TEMPERATURE CARBURIZATION TREATMENT ON THE HARDNESS BEHAVIOR OF Alsl316 AUSTENITIC STAINLESS STEEL

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ABSTRACT
There has been significant progress in the surface modification of stainless steel in order to enhance its surface hardness without major loss in other important properties. In this work, the possibility of enhancing the hardness property of austenitic stainless steel through a low-temperature pack carburization process was explored and investigated. Taguchi approach with L9 orthogonal array was used to optimize the hardness property of the carburized steel via the manipulation of the process parameters viz: carburizing temperature (350, 450, 550 ºC), carburizing time (8,16, 24hrs) and carburizer/energizer ratio (90:10, 80:20,70:30). The surface hardness profiles of the carburized layer were investigated. Taguchi analysis result shows that optimal hardness of 407Hv was achieved. The optimization of the carburized surface properties revealed that the carburizing temperature of 550 ºC, carburizing time of 24hrs and carburizer/energizer ratio of 90:10 are the prominent factors and levels for achieving optimal condition respectively. The models for optimal conditions were validated and the prediction adequacy are found to be within the limit of 2-6% prediction error, indicating the models are accurate and adequate to predict the responses. Optical microscope micrographs and morphologies indicates that the modified steel layer obtained at the optimal condition shows mild deeper case profile compared to non-treated stainless-steel samples. Equivalent of 104% improvement in the case hardness was achieved for the samples investigated with optimized condition. The level of improvement attained in the hardness therefore suggest that the ultimate objective of surface modification of austenitic stainless steel investigated in this study has been achieved.

Keywords: Carburizer, Energizer, Hardness, Low-Temperature, Treatment

INTRODUCTION
Stainless steels are widely used where corrosion resistance is of primary importance. The corrosion-resistant nature of stainless steel originates from the presence of the alloying element chromium, which forms a very stable passive layer that protects the steel. All stainless steels contain iron as the main element and chromium in amounts ranging from about 11% to 30%, in addition to 1% to 2% manganese. Chromium provides the basic corrosion resistance to stainless steel. A thin film of chromium oxide forms on the surface of the metal when it is exposed to oxygen in the air, acting as a barrier to further oxidation, rust, and corrosion (Liu *et al*., 2020). Additional research highlights that the stability and self-repairing nature of this passive layer play a crucial role in extending the material's service life, particularly in aggressive environments (Zhang *et al*., 2022).
Austenitic stainless steel (ASS) is undoubtedly the most widely used stainless steel, currently accounting for 70% to 80% of global stainless-steel production. Its composition ranges from 18% to 25% Cr and 8% to 20% Ni, making it fully austenitic and consequently non-magnetic. Adding Mo and other alloying elements further increases its outstanding resistance to general and pitting corrosion, enhancing its unique properties. Despite its advantages of good corrosion resistance in various acid conditions, this material suffers from low surface hardness and extensive wear, hindering its broader application in tribological settings such as control rods used in nuclear reactors, small bore weapon components, watch cases, tube fitting ferrules, piston rings, and pitch gears (Srinivasan, 2021). Recent studies have focused on improving the tribological performance of ASS by integrating novel surface modification techniques, such as plasma-assisted processing and advanced coating technologies, to enhance hardness and wear resistance (Chen *et al*., 2023).
The common practice for improving the surface properties of steels, particularly hardness and wear resistance, involves surface engineering designs, including thermochemical heat treatment (De la Rosa *et al.,* 2020). Traditional thermochemical treatments for surface engineering are typically carried out in carbon and/or nitrogen-bearing gases, usually maintained at temperatures above 900°C, commonly known as carburizing and nitrocarburizing. In carburizing treatment, wear and hardness improvements rely on the development of complex thin layers due to the diffusional reaction of carbon elements with the substrate. Austenitic stainless steel is usually subjected to conventional carburizing treatment but suffers at high carburizing temperatures as the diffused carbon reacts with the chromium in the steel to form carbides, thus depleting chromium from the solid solution. This depletion significantly affects corrosion resistance, leading to a well-documented trade-off between tribological and corrosion properties (Sura *et al*., 2017).
Recent research has explored alternative low-temperature carburization methods to mitigate chromium depletion while achieving desirable surface hardness and wear resistance. Low-temperature carburization, conducted at temperatures below 500°C, has shown promising results in retaining corrosion resistance while significantly improving surface hardness through the formation of an expanded austenite phase (Lopez *et al*., 2023). Therefore, this work attempts to enhance the surface properties of austenitic stainless steel 316 grade under a low-temperature pack carburization condition to obtain optimum properties via the Taguchi optimization technique. The findings of this study aim to contribute to the ongoing advancements in surface engineering techniques for

stainless steels, facilitating broader applications in high-performance and demanding industries.

MATERIALS AND METHODS

Samples preparation

The substrate material selected for this research is AISI 316 austenitic stainless steel, sourced from CENCO SAINS Scientific Supplies Malaysia. This material, received in a rod form with a length of 1000 mm and a diameter of 15 mm, was initially subjected to X-ray fluorescence (XRF) analysis to confirm its chemical composition (Dahmardeh *et al.,* 2020). The material was then cut and machined into standard hardness test samples using a CNC lathe machine, following the procedures outlined by Peng *et al*. (2018). Subsequently, all substrate samples were manually ground with silicon carbide emery paper to a 1200-grit finish to ensure a fine surface texture. After grinding, the samples were polished using 1 µm aluminum oxide paste on a velvet polishing machine. Finally, the substrates were thoroughly cleaned and degreased with soapy water and acetone solution, a standard method to eliminate surface contamination (Liu *et al*., 2019).

Substrate Surface Activation

All prepared substrate samples underwent surface activation treatment, where the samples were soaked in a concentrated hydrochloric acid (HCl) solution for 15 minutes to remove the passive oxide film commonly formed on austenitic stainless steel (Zhou *et al*., 2017). The removal of the passive layer facilitated the diffusion of carbon into the steel at higher temperatures, as observed by Kim *et al*. (2016). This treatment is essential to enhance the carburizing process, enabling a faster diffusion rate of carbon into the surface of the alloy by eliminating the protective oxide barrier (Xu *et al*., 2018).

Preparation of Wood Charcoal and Energizer Powder Mix

The wood charcoal was broken into pieces using a hammer and ground into fine particles via a ball milling machine, following the procedures outlined by Sari *et al*. (2017). The ground charcoal was sieved to achieve 1 to 2-mm particle sizes. Barium carbonate ($BaCO_3$) was selected as the energizer due to its ability to enhance the carburizing process (Shin *et al*., 2015). The carburizer/energizer mixture was prepared in weight ratios of 70:30, 80:20, and 90:10 (carburizer/energizer). This specific mixture composition is consistent with previous studies investigating the effects of carburizing compound ratios on the carburization of stainless steels (Goglia *et al*., 2019).

Low-Temperature Carburizing Treatment

For this investigation, a low-temperature pack carburizing process was employed to prevent the formation of chromium carbide, a common issue in high-temperature carburization processes (Zhou *et al*., 2018). Eighteen surface-activated substrate samples were loaded into a carburizing box containing a preheated mixture of charcoal and energizer. The box was sealed with clay to prevent carbon loss and unwanted gases from entering the furnace (Aydin *et al*., 2015). The carburizing treatment was carried out at temperatures of 350°C, 450°C, and 550°C for durations of 8, 16, and 24 hours, based on a Taguchi experimental design (Taguchi, 1986). The substrates were allowed to cool in the furnace to room temperature post-treatment.

Design of Experiments

The experimental design for this study was based on the statistical design of experiments (DOE) using the Taguchi method, with Minitab 17 software employed for analysis. The Taguchi approach is known for its efficiency in determining optimal factor levels while minimizing experimental cost (Phadke, 1989). The study followed an L9 orthogonal array design, with three factors: carburizing time, carburizing temperature, and carburizing compound each at three levels, and three responses hardness, wear rate, and coefficient of friction (COF). The factors and their corresponding levels are shown in Table 1, and the experimental layout is presented in Table 2, which follows the guidelines from previous work by Tohidi *et al*. (2017).

The summary of the factors and their levels selected for this experiment are given in Table 1. A total number of 18 samples were used for the carburizing experiment.

Table 1: L_9 Taguchi design for carburizing treatment parameters and their levels.

Factors	Factors code	Level		
		1	2	3
Carburizing Temperature	A	350	450	550
Soaking time (hrs.)	B	8	16	24
Wt% Carburizer/ Energizer	C	90:10	80:20	70:30

The factors and their levels chosen for conducting the experiment was considered based on exhaustive and critical review of past research works. The experimental layout of the control factors and levels are shown in Table 2 (coded values).

Table 2: L_9 Taguchi Orthogonal Array (3^3) Design Matrix (coded)

Experiment Runs	Control Factors		
1	1	1	1
2	1	2	2
3	1	3	3
4	2	1	2
5	2	2	3
6	2	3	1
7	3	1	3
8	3	2	1
9	3	3	2

Taguchi Experimental Analysis

The Taguchi method considers both controllable design parameters and uncontrollable noise factors (environmental influences) when optimizing product quality (Phadke, 1989). In this study, the Signal-to-Noise (S/N) ratio was used to analyze the data, accounting for both the mean and variability of the results (Cheng *et al*., 2018). For the hardness property, the larger-the-better (LTB) criterion was chosen as the quality characteristic for optimization, following similar methodologies from Nouri *et al*. (2016).

Samples Characterizations

The characterization of the carburized samples involved the analysis of microstructures and hardness properties. The carburized and untreated samples were analyzed to assess changes in microstructure post-treatment (Luo *et al.*, 2020).

Hardness Measurements

Hardness measurements were conducted using a Vickers microhardness tester on both treated and untreated samples. The testing procedure was based on the guidelines of ASTM E384 (2017). A minimum of five measurements were taken from different locations on the surface of each sample, and the average hardness value was recorded. Additionally, hardness depth profiles were performed on cross-sections of the treated samples to assess the carburized layer's hardness distribution, following similar techniques described by Lee *et al*. (2015).

RESULTS AND DISCUSSIONS

Chemical Composition Analysis

The chemical composition of as-received austenitic stainless steel is given in Table 4 below.

Table 3: Chemical Composition of AISI316 stainless steel

Element	C	Si	Mn	P	S	Cr	Ni	Mo
%Composition	0.0210	0.330	1.610	0.0360	0.0210	16.650	10.050	2.10

Surface Hardness of the Carburized Steel

The result obtained for the surface hardness of the carburized layer under low-temperature carburizing treatment can be seen in Table 4. The experiment was designed based on the Taguchis L9 orthogonal array, involving nine experimental trials with three factors at three levels.

Table 4: Hardness values for the carburized samples

Experimental runs	Control Factors			Hardness value (Hv)		Mean Hardness (Hv)
	A	B	C			
1	350	8	90:10	212	224	218
2	350	16	80:20	212	228	220
3	350	24	70:30	234	222	228
4	450	8	80:20	238	250	244
5	450	16	70:30	259	269	264
6	450	24	90:10	304	306	310
7	550	8	70:30	295	289	292
8	550	16	80:10	324	397	385
9	550	24	90:20	382	398	390

A: Carburizing temperature B: Carburizing Time C: carburizing compound

It can be observed from Table 4. that the maximum hardness of 390Hv was achieved at the carburizing temperature of 550 ºC, carburizing time of 24 hours and with the carburizing compound ratio of 90:10. This condition correspond to the sample treated at experimental trial 9. On the other hand, the sample that was carburized at carburizing temperature of 350ºC, carburizing time of 8 hours and carburizing compound ratio of 90:10 showed the least average surface hardness of 218Hv. This indicates that samples treated at very low carburizing temperatures (such as 350ºC) showed little or no carbon diffusion to the surface of the treated steel samples. Similar findings were reported by Cheon *et al.* (2024), who observed that low-temperature vacuum carburizing without surface activation led to limited carbon diffusion in AISI 316L steel.

Carburization Temperature and Time on the Surface Hardness of Carburized Samples

The plots of the surface hardness of the carburized steel samples with an increase in carburizing temperature and carburizing time were presented in Figures, (1-3).

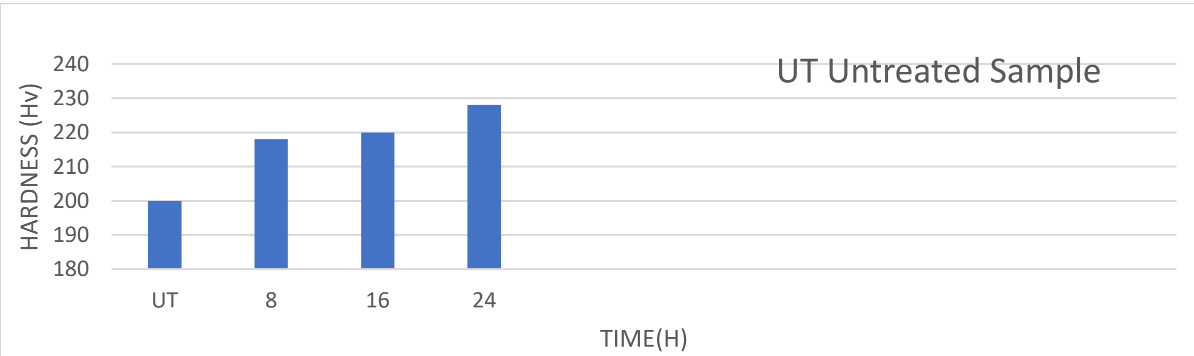

Figure 1: Surface hardness of samples carburized at 350ºC for 8 – 24 hours

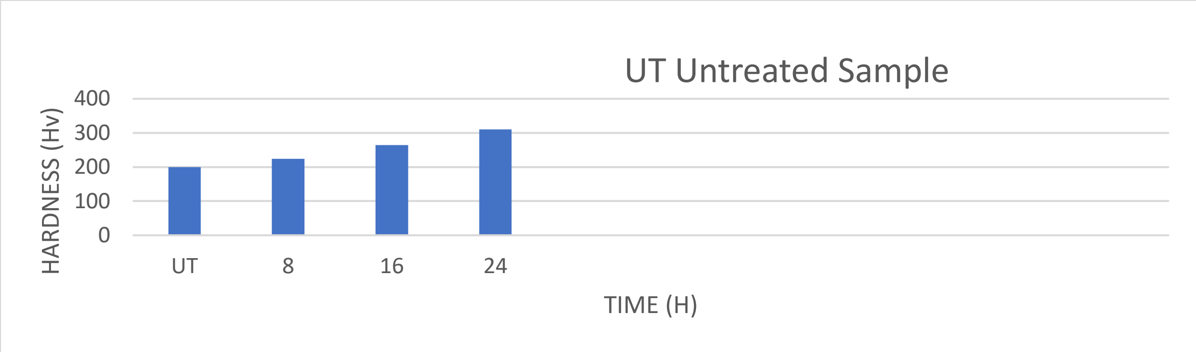

Figure 2: Surface hardness of carburized samples at 450 °C

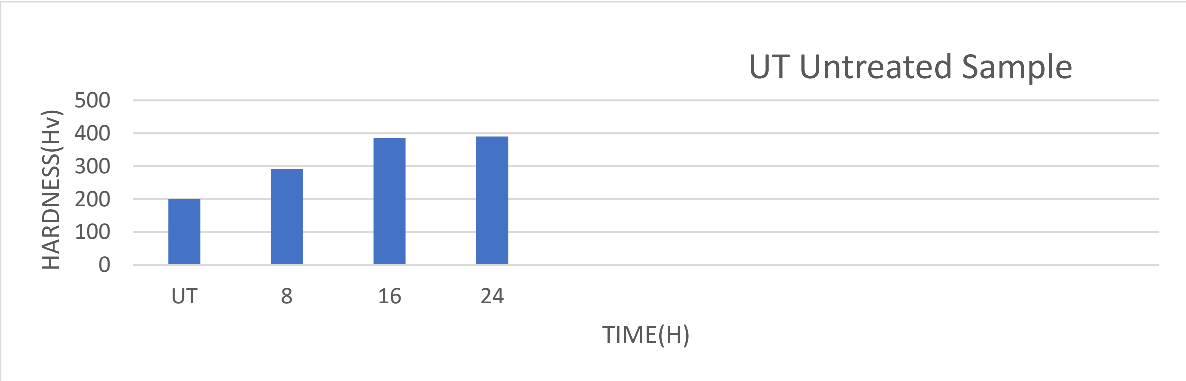

Figure 3: Surface hardness of carburized samples at 550°C

It was observed that the samples that carburized at 350 °C for the different carburizing times of 8, 16, and 24 hours (Figure 1), has little increase in the surface hardness. The sample treated at 350 °C for 8 hours shows the least surface hardness (Figure 1) compared with the sample treated at 16 and 24 hours with the surface hardness values 220HV and 228HV respectively. Samples treated at 350 °C for 8 hours have the least surface hardness of 218HV. The surface hardness slightly increased as the carburizing time increased up to 24 hours. The amount of carbon that diffused into the steel surface at 350 °C was too low to significantly increase the surface hardness.

Figure 2 shows the plot of hardness for samples that were carburized at 450 °C for the different carburizing times of 8, 16, and 24 hours. From the Figure, it can be seen that all the samples treated at 450 °C have higher surface hardness than the samples treated at 350 °C, Samples carburized at 8, 16, and 24 hours have surface hardness values of 244, 264, and 310HV respectively. This indicates that higher temperature and time lead to higher hardness. This trend aligns with the findings of Liu et al. (2020), who reported that low-temperature gaseous carburization significantly improved the fatigue performance and hardness of AISI 316L

The variation in hardness for the sample treated at 550°C for different carburizing times of 8, 16, and 24 hours is shown in Figure 3. This figure indicates that all samples tested under these conditions exhibited an increase in hardness compared to other treatment temperatures. Similar trends have been observed in previous studies on carburization, where increased process duration and temperature enhance hardness due to carbon diffusion (Baali *et al.*, 2023). The sample carburized for 8 hours attained a surface hardness of 292 HV, while the sample carburized for 16 hours reached 385 HV. A maximum surface hardness of 390 HV was achieved after 24 hours of carburizing, representing the peak hardness in this experiment. These results align with earlier research indicating that prolonged carburization improves carbon absorption and hardness (Dabbashi, 2023). This value was 100% higher than the substrate hardness, indicating moderate carbon absorption and surface carbon enrichment from the carburizing treatment (Ramadan, 2023). The improvement in surface hardness is likely due to increased carburizing temperature and time, which facilitates sufficient decomposition of the carburizing compound, a phenomenon well-documented in surface engineering literature (Aydin *et al.*, 2015).

Taguchi Optimization Analysis of the Carburized Steel Characteristics

Analysis of Surface Hardness of the Carburized Steel

The hardness characteristic of the carburized steel sample was analyzed using Taguchi S/N ratio based on the higher-the-better criterion. The experimental data were converted into S/N ratios using Minitab 17 statistical software.

Table 5: Signal to Noise (S/N) Ratio for The Surface Hardness of the Carburized Steel

S/N	Control Factors			Hardness (HV)	S/N ratio
	A	B	C		
1	350	8	90:10	218	46.7691
2	350	16	80:20	220	46.8485
3	350	24	70:30	228	47.1587
4	450	8	80:20	244	47.7478
5	450	16	70:30	264	48.4321
6	450	24	90:10	310	49.8282
7	550	8	70:30	292	49.3077
8	550	16	90:10	385	51.7092
9	550	24	80:20	390	51.8213

A: Carburizing temperature B: Carburizing Time C: Carburizing compound

Table 5 presents the signal-to-noise ratio for the surface hardness of the carburized steel samples. From the Table, it can be observed that the highest hardness of 390Hv attained at experimental run 9 (carburizing temperature of 550 ºC, carburizing time of 24 hours, and carburizing compound ratio of 80:20), gave the highest S/N ratio of 51.8213.

Table 6: Response Table for Signal Noise Ratios for hardness

Level	Carburizing temperature	Carburizing time	Carburizing compound
1	222.0	251.3	304.3
2	272.2	289.7	284.7
3	355.7	309.3	261.3
Delta	133.7	58.0	43.0
Rank	1	2	3

A: Carburizing temperature B: Carburizing Time C: carburizing compound

Table 6 presents the response table for hardness characteristics. The dominant control factors were identified using the delta statistics in the response table for S/N ratios, as shown in Table 5. The delta statistics were calculated based on the difference between the highest and lowest average values of each factor. Ranks were then assigned according to the delta values, with the highest delta value receiving the first rank, representing the most significant factor affecting hardness. Table 8 indicates that carburizing temperature, with a delta value of 133.7, is the most influential factor. The second most significant factor is carburizing time, with a delta value of 58.0, followed by the carburizing compound, with a delta value of 43.0.

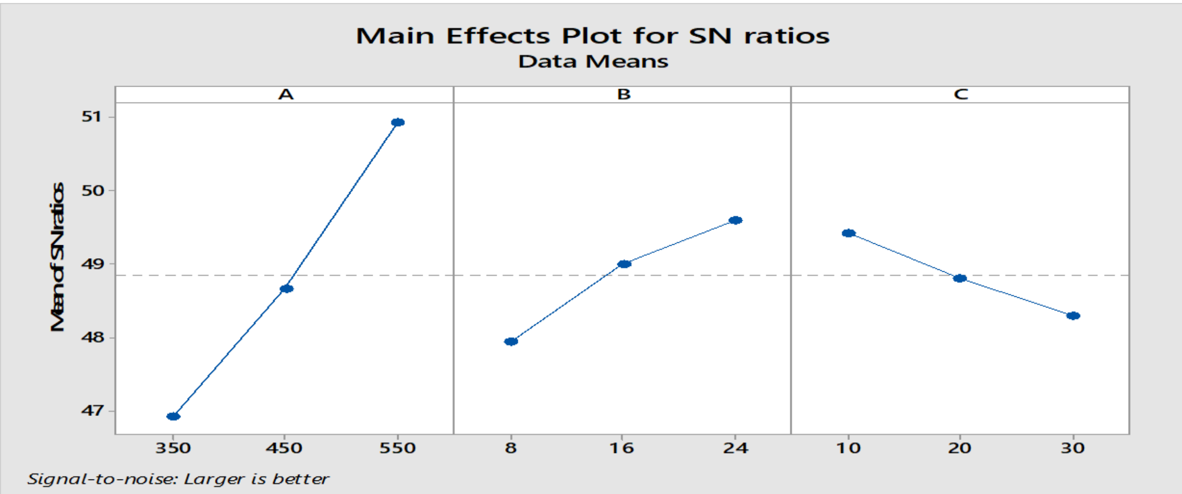

Figure 4: Main Effect Plot For S/N of Hardness

The main effects plot for S/N ratios was generated (Figure 4), showing that hardness is significantly influenced by an increase in temperature. As indicated in Table 6, hardness gradually increases with rising carburization temperature and time. This effect is likely due to the formation of carbon precipitate on the surface layer. The steep increase in S/N ratios for hardness, from 218HV to 390HV in Figure 4, supports the observation that the quality of the response improves as carburizing temperature and time increase. Therefore, the main effects plot for S/N ratios in Figure 4 suggests that A3, B3, and C1 (corresponding to 550°C, 24 hours, and a 90:10 ratio) are the optimal factor levels for achieving high S/N ratios and enhanced surface hardness.

Confirmation Tests for Hardness

Table 7: Results of the confirmation experiment for hardness of carburized samples

Initial Process Parameter		**Optimum Control Factor**	
		Prediction	**Experimental Values**
Levels	A3B3C2	A3B3C1	A3B3C1
Hardness	390	402	407
% Error	1.23		
% Improvement	104%		

A: Carburizing temperature B: Carburizing Time C: carburizing compound

A confirmatory test was carried out so as to validate the outcome of the analysis for the optimized conditions. The confirmation experiment was conducted by performing a new experiment based on the new optimized factor settings A3B3C1 to predict the hardness property. The prediction value was obtained from the Taguchi using Minitab 17 software. The result of the experiment was conducted under the factor level combinations of A3B3C1 and was compared with the prediction value as shown in Table 9. An error of 2.2% was observed indicating that the resulting Taguchi prediction was adequate to predict hardness characteristics to a reasonable accuracy.

CONCLUSION

Based on the result obtained, the low-temperature pack carburization treatment was successfully employed to modify the hardness characteristics of AISI316 stainless steel. The Taguchi analysis for single optimization of the low-temperature carburization process shows that the carburizing temperature and time are the most prominent factors influencing the hardness property. Also, the optimization of the modified surface responses shows that peak carburizing temperature (550ºC) and time (24hrs) gave optimum results for the hardness property. Taguchi's prediction for the optimal parameter settings was validated with a minimum error of 1.23%, indicating the adequacy and accuracy of the prediction. A maximum hardness value of 407HV was achieved for the sample carburized at the optimized condition (A3B3C1), which is a 104% improvement as compared to that of the as-received substrate (200Hv). The optimum thickness layer and deeper case profile were obtained at the optimum carburizing condition of a carburizing temperature of 550 °C, carburizing time of 24 hours, and the carburizing compound ratio of 80:20.

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