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Carburization-Based Optimization of AISI 8620 Steel Using Rice Husks and Charcoal as Carburizers

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Abstract. This study was centered on improving the mechanical properties of AISI 8620 steel using the carburization technique. The failure in service conditions of many steel components such as cams, gears, and shafts necessitated the research as it demands that they possess both high wear-resistant surfaces and tough shock-resistant cores. Standard test samples prepared from the steel material were subjected to a pack carburization process using rice husk and charcoal as carburizers, and the energizer – calcium trioxocarbonate (IV) at temperatures of 800, 850, 900, and 950 °C, and held for 60, 90, and 120 minutes. The samples were quenched in water and tempered at 500 °C for 60 minutes. After the pack hardening process, the test samples were subjected to tensile, impact, and hardness tests. From the data obtained, ultimate tensile strength (UTS), Hardness, Young's Modulus, engineering strain, and impact strength were calculated. The case and core hardness of the carburized samples were noted, and an optical microscope was used to observe the microstructural features of the case-hardened, quenched, and tempered samples. The responses (mechanical properties of steel) were optimized using response surface methodology to obtain the optimum carburizing conditions-temperature and holding time. Results showed that the sample's microhardness core and microhardness case increased from 253 to 327 HV and from 243 to 339 HV as the holding time increased from 60 to 120 minutes, indicating an appreciable increase in the mechanical property of the samples. The optimum carburizing conditions were at a temperature of 885 °C and a holding time of 120 minutes. Hence, the carburization of AISI 8620 steel using rice husk and charcoal as carburizers improved the steel material's case, core, and mechanical properties.

Keywords: materials science, carburization, rice husks, charcoal, steel, optimization.

1 Introduction

Carburization is one of the case hardening methods in which carbon layer formation is induced on the surface of a substance in order to improve the strength and hardness properties. Carburizing temperature boundary is within 850-950 °C. Carburization is employed on materials that cannot be hardened appreciably by hardening process, like low carbon steel. Hardenability is a property of steel that measures the depth and distribution of hardness obtained by quenching from the austenizing temperature. The ratio of transformation of austenite to martensite is the deciding factor for hardenability. Steel with high hardenability forms thick layers of martensite. In many engineering applications, it is desirable that steel being used should have a good surface hardness value in order

to resist wear and tear. The hardened surface is required to possess a soft and tough core to aid in shock absorption process. Surface hardening processes other than carburizing are: Cyaniding, Nitriding, induction hardening and flame hardening.

Steel materials with high carbon content of around 0.8 % are hard, but brittle, and therefore cannot be used in machine parts such as gears, sleeves and shafts that are exposed to dynamic bending and tensile stresses during operation [1]. A steel material with carbon content as high as 1.0 % is very hard to machine by cutting operations such as turning or drilling [1]. These concerns are solved by using low carbon steel materials such as AISI 8620, AISI 1020, etc. and subjecting them to some surface modification operations like carburizing, boronizing, nitriding, ion nitriding, etc. In addition, most

of the failures of some mechanical elements or components subjected to cyclic motions are caused by fatigue, as such, the fatigue performance of materials has prime importance for the design of structures which are subject to cyclic loading [2]. The surfaces of machine parts or components are the most susceptible zones to fatigue and tribomechanical failures, and fatigue cracks are generally initiated at the surface [3]. Therefore, there is a high incremental stress profile generation and distribution across the surfaces of machine parts under cyclic loading or a tribological condition. Hence, strengthening of the surface materials of such machine part or component is very imperative in enhancing fatigue life and tribomechanical characteristics of the material. The application of mechanical or thermo-chemical surface treatment like case carburizing, boronizing, etc. strengthens the surfaces of the steel material and in-turn initiates residual compressive stresses at the surface that would hinder fatigue crack development and propagation.

Therefore, this study was centered on the determination of the carburizing potential of rice husks and charcoal, as an agricultural residue, to provide carbon to the surface of a specimen of AISI/SAE 8620 mild steel when it is heat treated by pack carburizing. The consequent effects of carburizing operation on the tribo-mechanical properties of AISI 8620 steel were also studied. The optimum carburizing conditions (temperature and holding time) that yielded the best response characteristics of the carburized sample were obtained using optimization technique.

2 Literature Review

The determination of the effects of carburizing conditions (temperature and holding time) and the carburizing substances on the tribo-mechanical properties of the base material has been a research focus. These factors and their various combinations propose different mechanical properties and carbon-inducement percentage on the surface layer of the material- low carbon steel. Based on this, studies have been carried out in quest of improving the surface hardness of low carbon steel material and the determination of the consequent effects of the process. To this effect, Oluwafemi et al [4] used palm-shell as carburizing substance to pack carburize AISI 1020 steel. They used a treatment temperature ranging from 800 to 950 °C and holding time of 60, 90, and 120 minutes. The result obtained showed a microstructural pattern of a hardened steel specimen, thereby attesting that there was an inducement of carbon on the surface. Also, Istiroyah et al [5] employed charcoal gotten from coconut shell and rice husk at a temperature of 600°C to carburize AISI 316L steel. They use a treatment temperature of 400 °C and a soaking time of 480 minutes. The results obtained proved that carbon distribution in the steel carburized with coconut shell was better than that of rice husk. In addition, Siti et al [6] studied the effects of paste carburizing treatment on mechanical properties of ASTM A516 low carbon steel. Tensile test, hardness test (Rockwell) and microstructural examination were conducted on the carburized sample. The paste carburizing treatment was carried out at temperature of 700, 750, and 800 °C for 6 hours holding

time. The results depicted that paste carburized samples provide significant improvement on both tensile strength and hardness values compared to uncarburized samples. This was associated with the formation of hard carburized surface-layer on the substance. Increasing the carburizing temperature profoundly improved both hardness and tensile strength, as the results of deeper carburized layer produced. Paste carburizing was found to induce formation of carburized layer at shorter time and lower temperature compared to pack carburizing method.

3 Research Methodology

3.1 Material description

The base material employed in this study was AISI 8620 steel and pack carburizing method was used in assessing the surface hardening potential of an agricultural residue- rice husk and charcoal.

The steel material was sourced locally and was analyzed using atomic absorption spectrometer (AAS). It was cut and machined to standard test sample sizes according to American Society for Testing and Materials (ASTM) standard specifications using a lathe machine. Carburization was done using heat treatment furnace. The tensile test was conducted using Computer Controlled Electro-Hydraulic Servo Universal Tensile Testing Machine (Model HLCS-600). Metallurgical microscope (Model Olympus PMG-3) was used to study the microstructures while micro-hardness testing machine (Model UH930) was employed to measure the micro-hardness while impact testing machine (Model UI820) was used to ascertain the impact strength of the steel material.

The method employed in this study includes: the determination of the percentage composition of the locally sourced AISI 8620 steel material, elemental analysis (%) carburizing materials, pack carburizing method application, design of experiment, mechanical tests and optimization.

3.2 Chemical analysis of AISI 8620 steel

AISI 8620 steel was sourced locally and analyzed using atomic absorption spectrometer (AAS). It was cut and machined to standard test sample sizes according to American society for testing and material (ASTM) standard specifications using a lathe machine. The AISI 8620 steel was polished with a polishing machine for it to bring out the best results. It was placed in the spectrometer machine and spark was introduced to give the result shown in Table 1.

Table 1 – The compositional build of AISI 8620 steel sample

Elements	C	Si	Mn	Mo
Average content	0.200	0.165	0.750	0.158
Elements	Cr	Ni	Fe	P
Average content	0.450	0.470	97.886	0.0295

The sparking was done two to three times after which the average was taken.

3.3 Chemical composition of carburizing materials

The chemical compositions of rice husks were obtained through the following procedures: samples of rice husks were handpicked and washed clean to avoid impurities and sand. The rice husks were chemically treated with sodium hydroxide (NaOH) solution, neutralized with acetic acid and finally washed with distilled water and sun-dried. 2 kg of rice husks was measured and 1 kg of charcoal in the proportion of 7:3.

A grinding machine was used to reduce the size of the rice husks and charcoal individually and sieved to a standard sieve size of 450 μm to get uniform size. Mixture of rice husks/charcoal were melted and sparked in an extrusion machine at temperature of 120-150 $^{\circ}\text{C}$ and screw speed of 50 rpm to obtain the chemical analysis of rice husks/charcoal. The chemical compositions of the carburizing materials are shown in Table 2.

Table 2 – Chemical composition of the carburizing materials

Material	Elemental Analysis (%)				
	C	H	O	N	S
Rice husk	41.13	3.37	35.3	0.33	–
Charcoal	72.31	2.43	–	1.16	1.05

3.4 Pack carburization technique

Pack carburizing method was employed in this study. Carbon powder (derived from the carburizers) was mixed properly with an energizer (calcium carbonate) in the proportion of 7:3 after which the first set of samples was buried in the mixture inside a rectangular steel box. Clay mixed with Bentonite and moderate water was used to seal the rectangular steel box tightly to prevent carbon (11) oxide from escaping and in turn not allowing unwanted furnace gas from re-entering the steel box. The steel box was then charged into the furnace and allowed to heat to temperatures of 750, 800, 850, and 900 $^{\circ}\text{C}$, respectively. At each temperature the test specimen was soaked for one, two and three hours respectively according to the experimental design. The steel box was removed from the furnace with the help of tongs at each temperature and then the specimen was quenched in water at ambient temperature. The mixture configurations of the applied carburizers in the development of the test samples are shown in Table 3.

Table 3 – Mixture composition for pack carburizing operation

Mixture code	carburizing material	Amount of, g	Amount of energizer CaCO_3 , g
MC1	charcoal	500	–
MC2		500	333.3
MC3	rice husk	500	–
MC4		500	333.3

3.5 Design of experiment

The Design Expert 11.0 software was employed for the experimental design. In quest of finding the optimum factor combination that would yield the best response

characteristics of carburized AISI 8620 steel, response surface methodology tool was applied.

The input factors were temperature ($^{\circ}\text{C}$) and holding time (minutes). The response variables employed in the experimental design were ultimate tensile strength (MPa), strain (%), modulus of elasticity (MPa), impact strength (J), micro hardness case (H), and micro hardness core (H).

A total of 12 runs/simulations were obtained from the experimental design and were meticulously followed while performing the experiment. The mathematical inequality used in designing the experiment is described thus: temperature – in a range of 800-950 $^{\circ}\text{C}$; holding time – 60-120 minutes.

The ultimate tensile strength, strain, modulus of elasticity, impact strength and hardness (case and core structures) of pack carburized steel samples all determined using the appropriate machines. The microstructural characteristics of the produced samples of carburized steel were ascertained by first polishing the samples and etching them with 2 % Nital solution.

4 Results and Discussion

4.1 Microstructural analysis

Figure 1 a reveals the optical micrograph of the base (non-carburized) AISI 8620 steel material used.

It depicts steel in the state of supply. The structure observed is a matrix of ferrite and pearlite which are the characteristic constituents that yielded the chemical composition of the material steel as indicated in Table 1.

Figures 1 b-e correspond to the specimens that is heat treated using the carburizing mixtures indicated in Table 3. The figures show from the core to the surface, a progressive increment in the amount of pearlite. This amount remaining in the core equals the amount observed in the image of the specimen without heat treatment (Figure 1 a). This progressive increment in the proportion of pearlite shows that both the rice husk and the charcoal allowed the introduction of carbon (diffusion) into the steel specimen, showing an increment in the amount of carbon until certain distance from the surface (case depth). The case depth, as observed, was greater in the specimens that contained energizer material in its composition (Figures 1 c, e).

In this way, as stipulated in the literature [7] is contrasted with respect to the potentiating effect of the energizer in the increase of the diffusion rate of the carbon in heat treatment of pack carburizing. The results also showed that the carbon diffusion was more intense in the specimens that were heat treated with charcoal (Figures 1 b, c), the expected result, taking into account that the percentage of carbon in this, was higher by 43 % to the percentage present in the rice husk. In addition, it can be seen that in the specimens that were carburized with charcoal (MC1 and MC2) a greater percentage of carbon was obtained in this zone. Although, in the Figures 1 b, d, no uniform thickness of the diffused carbon was observed compared to Figures 1 c, e. It does not indicate that the carbon content did not increase at the surface of these specimens, it is sufficient to compare with Figure 1 a for a clearer view of the increase in the carbon contents of these specimens.

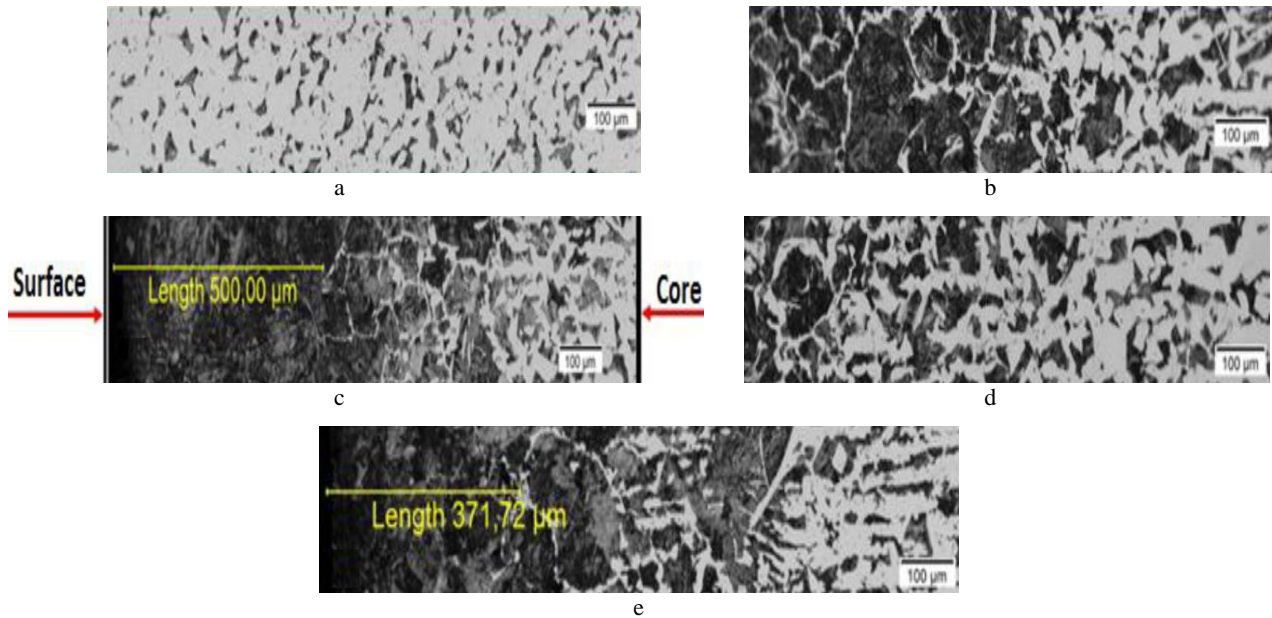


Figure 1 – Optical micrograph of AISI 8620 mild steel specimen: a – uncarburized base material; b – carburized specimen with MC1; c – carburized specimen with MC2; d – carburized specimen with MC3; e – carburized specimen with MC4

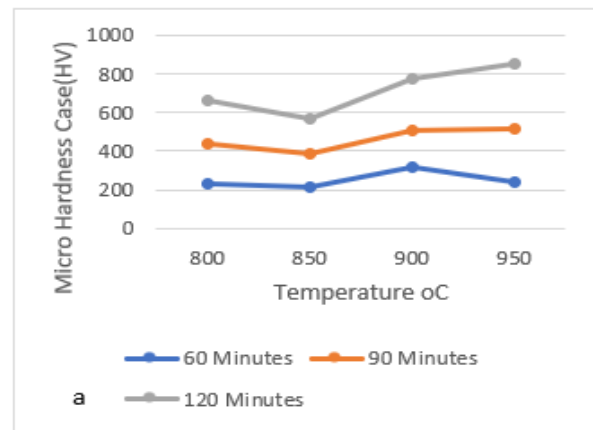
4.2 Core micro hardness

Figure 2 depicts the variations of micro hardness of the carburized steel at its case and core parts under the effects of carburizing temperatures and holding time.

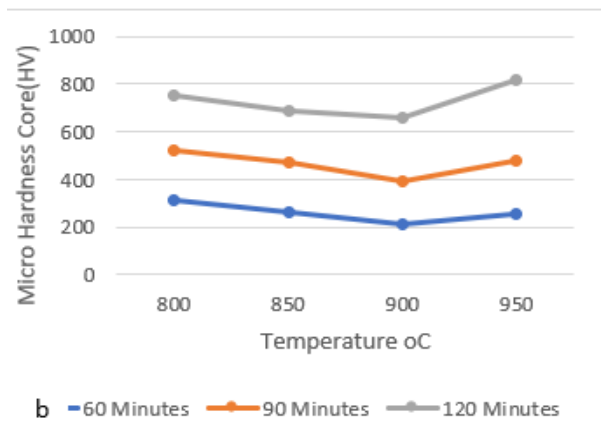
From Figure 2 a, it can be clearly seen that for all the holding time (60, 90, and 120 minutes), the micro hardness of the case dropped between carburizing temperatures of 800 to 850 °C and increased between temperatures of 850-900 °C. This was as a result of the distortion produced in the atomic structure of the samples and the material's adjustment to accommodate the treatment induced on it; as such, the micro hardness of the case was expected to drop at the earlier stage of the carburizing operation and rise after the atomic adjustments of the material in order to accommodate the treatment. Sequel to that, the inducement of some carbon percentage which led to the observed increase in micro hardness of the sample affirmed the explained distortion and adjustment of the atomic structure of the sample.

Also, a decrease in the case hardness of the sample was observed at a holding time of 60 minutes between temperatures of 900-950 °C while a continuous rise in the hardness of the case occurred at holding time of 90mins and 120 minutes, with a peak value reached at 120mins and 950 °C. This was so because, high carburizing temperatures allows for high hardness values of samples. The material needs to be transformed completely to austenite owing to its great affinity for carbon. Such if achieved, causes a large amount of carbon percentage to be induced into the material and consequently leads to the actualization of higher hardness values of the material.

Figure 2 b revealed the effect of carburizing temperature and holding time on the core micro hardness of the samples. From Figure 2 b, it was observed that all the samples carburized at 800, 850, and 900 °C followed the same trend beginning from high hardness value at the core when soaked for 60 minutes (316 HV) and decreased at 90 minutes holding time (207 HV), but increases as the holding time is increased to 120 minutes (230 HV).



a



b

Figure 2 – Effect of carburizing temperatures and holding time on the case micro hardness (a) and the core micro hardness (b)

Thus, attaining the respective peak hardness values at 120 minutes holding time. The uniform fall in the values of micro hardness of the core for holding time of 60, 90, and 120 minutes could be attributed to the distortions and adjustments of large volumes of atoms present at the core

of the samples which required an increased condition of carburizing operations (temperature and holding time) before accommodating the treatments induced into it.

Sequel to that, owing to the large volume of the core, the transformation of the material completely to austenite would take longer time and treatment temperature compared to the case. Increased hardness of the core occurred after its continuous decrease to the temperature of 900 °C. Beyond 900 °C, a uniform rise of micro hardness of the core was observed because of the

transformation to Austenite which has great affinity for carbon.

4.3 Ultimate tensile strength, engineering strain, and Young's modulus

Figure 3 shows the effects of carburizing temperature and holding time of the ultimate tensile strength and strain properties of AISI 8620 steel material.

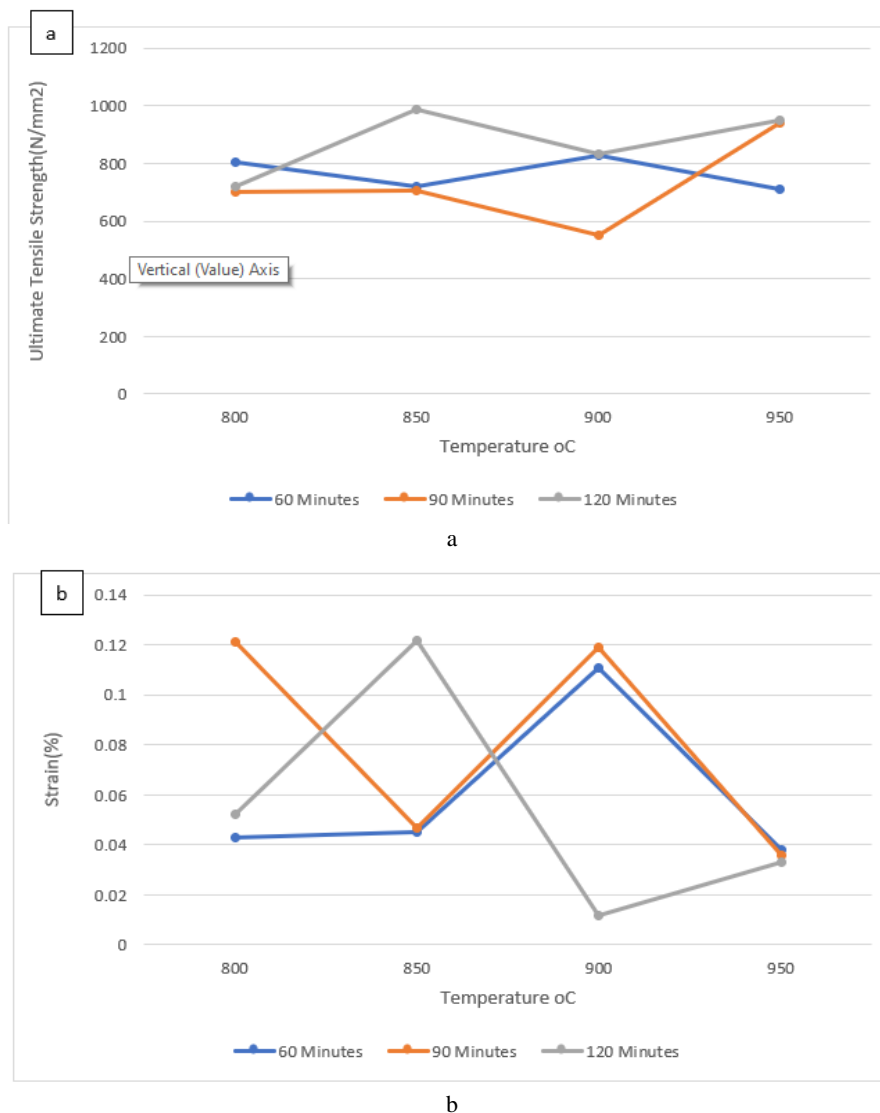


Figure 3 – Effect of carburizing temperatures and holding time on the ultimate tensile strength (a) and the engineering strain (b)

From Figure 3 a, the highest value of ultimate tensile strength was obtained at a carburizing temperature of 850 °C and holding time of 120 minutes. This was followed by carburizing conditions of 950 °C temperature and 90 minutes holding time. The lowest value of ultimate tensile strength was gotten at a carburizing temperature of 900 °C and holding time of 90 minutes. These observed characteristics of the material could be attributed to the distortion and rearrangement of the material's atoms during the carburization of operation.

In addition, Figure 3 b depicts the influence of carburizing temperature and holding time on the strain property of the steel specimen. From Figure 3 b,

maximum strain values of the material were obtained at carburizing temperatures and holding time of: 800 °C for 90 minutes, 850 °C for 120 minutes, and 900 °C for 90 minutes.

These notable strain characteristics of the material delineate high ductility parameter of AISI 8620 steel at the stated carburization conditions. Moreover, the lowest strain values were obtained at carburizing temperatures and holding time of: 800 °C for 60 minutes, 850 °C for 90 minutes, and largely, 900 °C for 120 minutes. Therefore, the material has low ductility property at these carburization conditions.

Figure 4 depicts the effects of carburizing temperature and holding time on the impact strength and elastic

modulus of AISI 8620 steel material respectively.

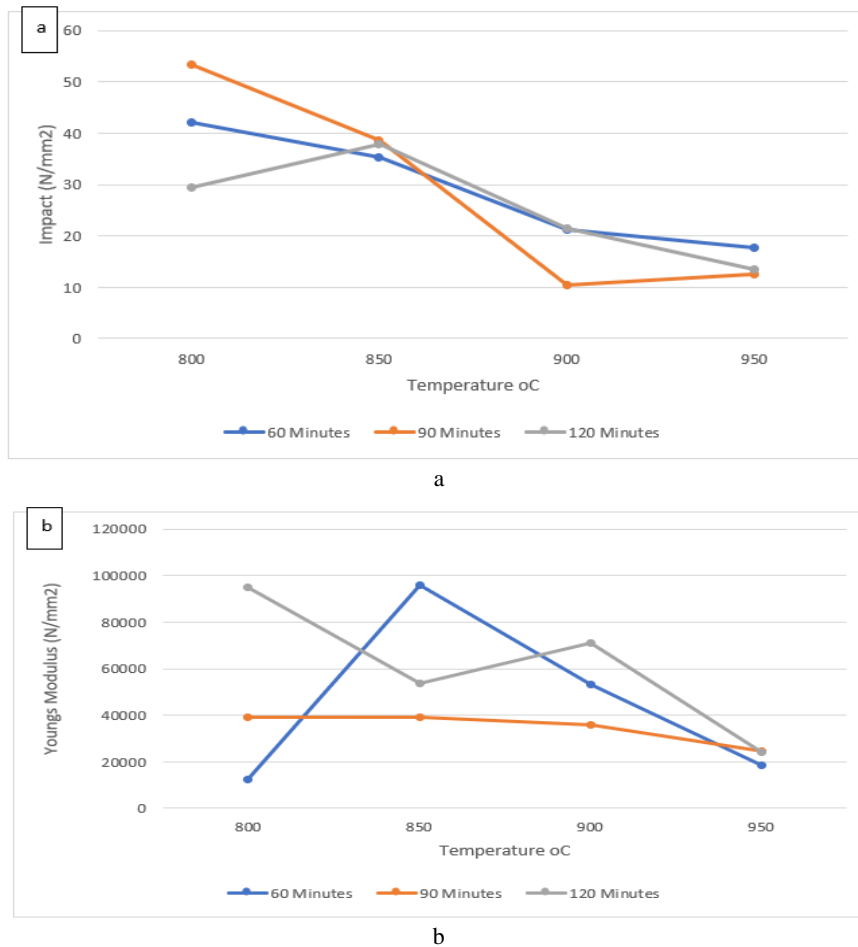


Figure 4 – Effect of carburizing temperatures and holding time on impact strength (a) and Young’s modulus (b)

From Figure 4 a, the highest value of impact strength was obtained at a carburizing temperature of 800 °C and holding time of 90 minutes. Also, the lowest value was gotten at a temperature of 900 °C and holding time of 90 minutes.

In addition, Figure 4 b presents the lowest value of elastic modulus achieved at a carburizing temperature of 800 °C and holding time of 60 minutes. While higher values of elastic modulus were obtained at 800 and 850 °C carburizing temperatures for holding time of 120 and 60 minutes, respectively.

These behavioural characteristics of the steel material could be explained through the study of iron-carbon phase diagram for the steel specification as reported by [8].

4.4 Optimization of the responses

The optimum operating temperature and holding time that would yield the best response values were obtained using numerical optimization technique.

Figure 5 shows the plot of desirability against temperature and holding time.

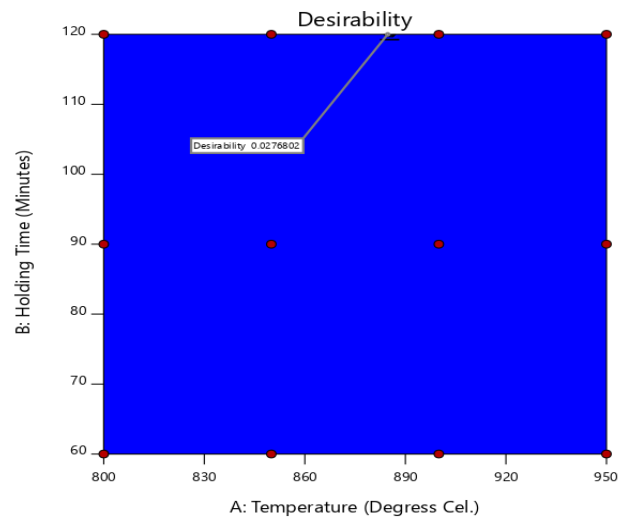


Figure 5 – Desirability for holding time against temperature

From Figure 5, the highest desirability value of 0.028 was obtained for factor combination of temperature at 885 °C and holding time of 120 minutes.

In addition, Figure 6 shows the contour plot of the response variable- ultimate tensile strength having the holding time plotted against the temperature.

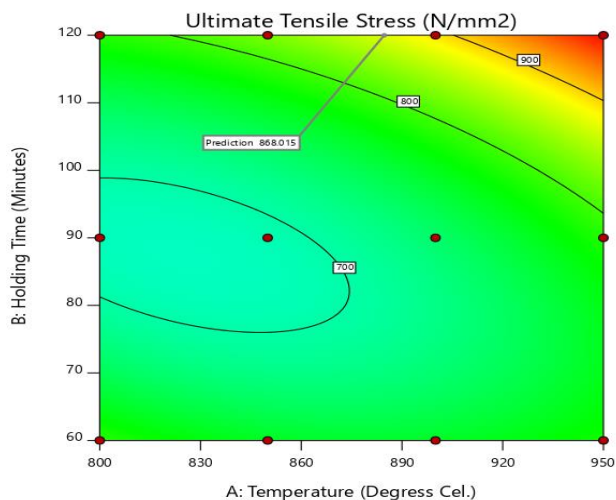


Figure 6 – Ultimate tensile strength for holding time against temperature

From Figure 6, it can be deduced that at the optimal factor combination of temperature 885 °C and holding time of 120 minutes, the predicted value of ultimate tensile strength of carburized AISI 8620 was 868 MPa.

This predicted value of ultimate tensile strength is well suited when matched with the other values observed from Figure 3 a.

Figure 7 shows the contour plot of the strain induced on the material at the optimal points of temperature and holding time, 885 °C and 120 minutes, respectively.

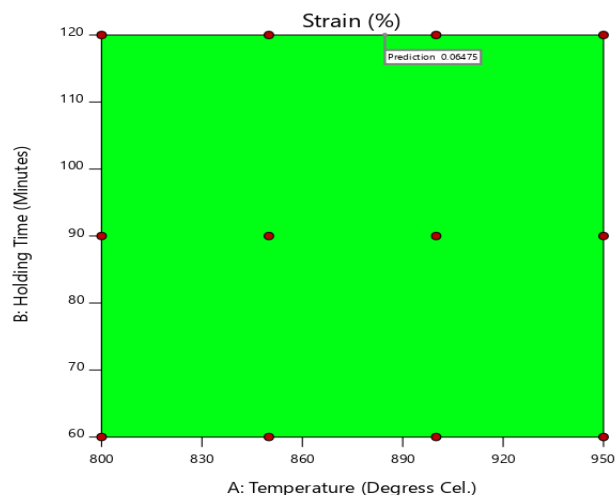


Figure 7 – Strain for holding time against temperature

From Figure 7, it can be inferred that the optimum holding time and temperature for strain minimization in carburized AISI 8620 were 120 minutes and 885 and 725 °C, respectively.

A strain value of about 0.06 % at these optimum factor combinations was predicted. An induced strain value of 0.06 % is good when compared with others shown in Figure 3 b.

Figure 8 shows the contour plot of Young’s modulus for holding time and temperature.

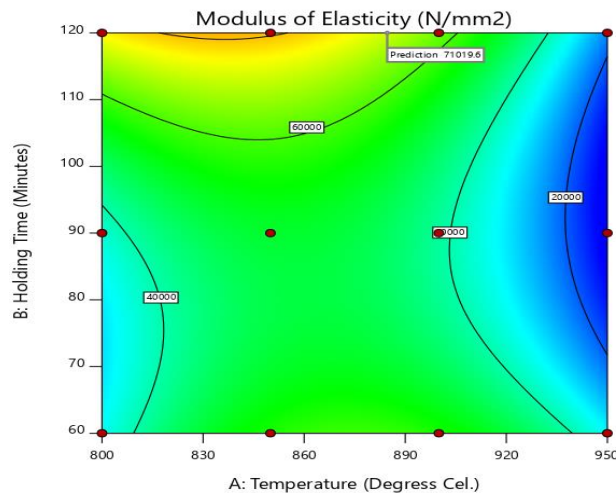


Figure 8 – Holding time against temperature

From Figure 8, it is evident that the optimum factor combination for a good yield of the response parameter-Young ’ s modulus is at a temperature 885 °C and holding time of 120 minutes. A response prediction of $7.1 \cdot 10^4$ MPa was obtained at this optimum point or design point.

Figure 9 shows the contour plot of the response parameter, impact strength. It attests to the optimum factor combination of temperature 885 °C and holding time of 120 minutes. The impact strength at this design point was 24.1 J.

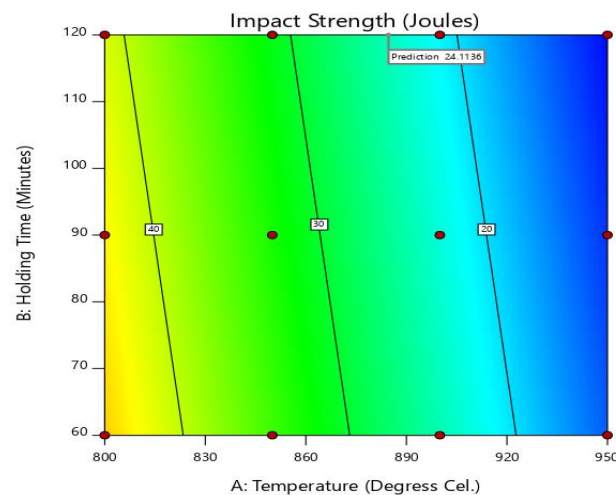


Figure 9 – Impact strength for holding time against temperature

Figure 10 shows the contour plots of the responses: micro hardness case and micro hardness core. From Figure 10 a, the optimum or design point for obtaining the best value of the response was at the factor combinatorial point of temperature 885 °C and holding time of 120 minutes. A predicted value of micro hardness case about 241.3 H was gotten at this design point.

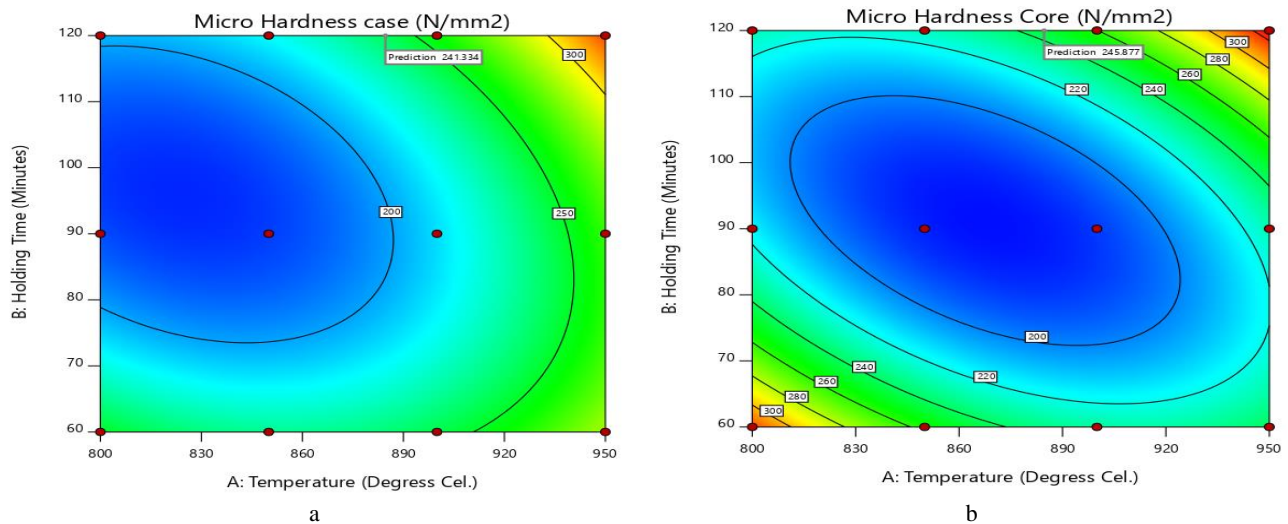


Figure 10 – Micro hardness case (a) and core (b) for holding time against temperature

Furthermore, Figure 10 b gave the optimum value of micro hardness core of carburized AISI 8620 of 245.9 H at the optimal or design point. This value is well suited when compared with others as shown in Figure 2 b.

Therefore, at the gotten optimal factor combination, the overall best response characteristics was obtained. The summary of optimal factor combination for better response characteristics is shown in Table 4.

Table 4 – Summary of optimal factor combination (temperature of 885 °C and holding time of 120 minutes)

Response	Mean	Median	StdDev	SE Mean	95 % CI low	95 % CI high	95 % TI low	95 % TI high
Ultimate tensile stress	868.015	867.950	132.433	81.504	668.517	1067.38	56.1749	1679.73
Strain	0.06475	0.06475	0.04082	0.0118	0.03881	0.0907	-0.11945	0.24895
Modulus of elasticity	71019.6	71019.6	25147.9	15477.0	33148.8	108890	-83130.2	225169
Impact strength	24.1136	24.1136	7.52327	3.45442	16.2992	31.9281	-14.1004	62.3277
Micro hardness case	241.334	241.334	43.1072	26.5298	176.417	306.25	-22.9017	505.569
Micro hardness core	245.877	245.877	16.4321	10.1129	221.132	270.623	145.153	346.602

5 Conclusions

The mechanical properties of low carbon steel material are improved by carburization process through the influence of the carburizing factors- carburizing temperature, carburizers and holding time. More so, from this study, the three carburizers employed in the process in the ratio of 75 % wt. : 25 % wt. strongly influenced the mechanical properties of the low carbon steel material.

The optimum conditions for carburizing low carbon steel material (AISI 8620) for good response characteristics are: temperature of 885 °C and holding time of 120 minutes as obtained from the numerical optimization performed. In addition, the presence of carbon-enriched skin (austenite to martensite) in the test samples can be inferred from the microstructural change and the good increase in hardness in most of the samples.

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