An- Najah National University

Faculty of Engineering

Chemical Engineering Department

Graduation Project (2)

Enhancement of Mechanical Properties of Mild Steel via Carburization Process using Residual Carbon Black

Prepared by:

Ghofran Nader Mohammad Ata

Mosheera Mohammad Saleh Jazi

Raghad Adnan Hasan Manasra

Supervisor:

Dr. Amjad El-Qanni

Co-supervisor:

Dr. Ihab H. Alsurakji

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Abstract

Car tires are the most solid waste that is produced globally every year. Nowadays, there is a tendency to improve the economic value of tire wastes as a source for producing several products, such as carbon black (CB), by pyrolysis process. In this research, the CB was used as a carbon source in pack carburization for mild steel (0.139 %C). So, the effect of the carburization temperature and holding time were studied on the hardness and fatigue characteristics of mild steel by carburizing it with 90% of CB as carburizer and 10% of Na_2CO_3 as an energizer, after that, it was quenched in water, then tempered at 550 °C for 1 h. In the first, the mild steel specimens were carburized at different temperatures (850, 900, and 950 °C) for constant time (2 h), the results showed that the surface hardness of carburized mild steel increased with increasing temperature. Then the effect of carburization time was studied at a constant temperature of 950 °C, the different times were 1.5 and 2.5 h, in addition to 2 h. Also, the surface hardness of carburized mild steel has been increased with increasing the soaking time. Besides, the fatigue life of carburized mild steel has been improved almost 10 times than untreated specimens. For the sake of comparison, commercial charcoal was used as a carbon source for carburizing mild steel at 950 °C for different times (2 and 2.5 h). The surface Vickers hardness of carburized steel by charcoal (820 HV) was better than carburized steel by CB (564.1 HV), however, the hardness of untreated specimen 260.5 HV. For the fatigue life for carburized specimens by charcoal, they need further study by re-conducting their experiments and tests as they differ in their results from what is found in the literature.

Keywords: Mild steel; residual carbon black; pyrolysis; pack carburization; hardness; fatigue resistance.

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Acknowledgments

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1. Introduction

Tires are the most products produced in modern society, for their great role in the transportation process, where billions of tires are produced globally every year. At this time, a huge quantity of tire wastes was disposed in landfills, which requires large areas of land to fill and pile in, wasting land resources in addition to threatening the environment after long-term storage. Therefore, it is important to find a sustainable method to get rid of used tires and exploit them for beneficial matters that are not harmful to the environment. Alongside gasification and incineration methods that are used to produce energy from these solid wastes at very high temperatures, pyrolysis is one of the most common processes used due to its economical viability as it works at lower reaction temperatures and some commodity products are recovered from waste tires, such as pyrolysis oil, pyrolysis gas, and solid coke (Zhong et al., 2020).

Iron and steel are based materials in the industry that are used in the construction of roads, railways, buildings, and the largest modern structures such as stadiums, skyscrapers, bridges, and airports are supported by a steel skeleton. Even steel was employed for reinforcing concrete structures, and it is construction materials for nails, bolts, household products, cooking utensils, and other common applications including shipbuilding, pipelines, and mining (Ochshorn, 2002). So, the improvement of mechanical properties to meet desired engineering applications has been a key point in steel research for centuries. Carburization is the main process used in industry for such purposes, especially in Palestine. The carburizing steel is widely used as a material of machines, gears, springs, automobiles, and wires which are required to have threshold standards for strength, toughness, hardness, and fatigue resistance (Pandaa et al., 2014).

Hence, the main objective of this project aims at studying the effect of the residual solid coke, hereinafter called carbon black (CB), as a carbon source in the carburization process for mild steel through addressing the effect of carburization temperature and time on the mechanical properties of it which are hardness and fatigue.

2. Constraints and Standards:

Constraints: the composition test of specimen is required to know the carbon content of it before and after the carburization process, however, it is not available at our university. So, a search was made for specializing companies in iron and steel work. Al-HADDAD INVESTMENT & STEEL CO (Hebron, West Bank) was found specializes in the manufacture of several types of steel, and the composition test with the Optical Emission Spectrometer (OES) is the main test for it in production processes. Thus, after the request was approached, the agreement was conducted by supporting this project by performing a composition test, in addition to supplying the required steel type specimens.

Standards: the Vickers Hardness test was applied as standard test (ASTM E92-17).

3. Literature Review

There are several different types of heat treatment processes that are used to modify the surface and structural properties of engineering components, especially steel, which is a series of timed heating and cooling (Murugan and Mathews, 2013). The suitable heat/thermal treatment method should be chosen based on the desired properties of steel for the required designed applications (Senthilkumar and Ajiboye, 2012). Case hardening is widely used in applications where a hard surface is required in combination with core toughness to resist fracture from impact loading (Bepari, 2017). Carburizing is a way of a case hardening process in which carbon diffuses into the surface of low carbon steel (Abdullah et al., 2017). This treatment consists of heating the piece of metal to austenite phase temperature (800 °C - 1000 °C) for a certain period, in a medium that provides carbon (Abdullah et al., 2017). Case hardening by carburizing consists of three separate processes, carbon enrichment process for steel surface, quenching to hardening of the components, and tempering to provide ductile and tough core (Maisuradze and Kuklina, 2018). The carburization process is carried out for metal that cannot be hardened in the direct heat treatment process and the reason for this is the low strength of carbon content (Murugan and Mathews, 2013). The most affected factors in the carburization process are the carbon potential, carburizing temperature, soaking time, and the quenching media (Akanji et al., 2015). Figure 1 shows the schematic representation of the carburizing process.

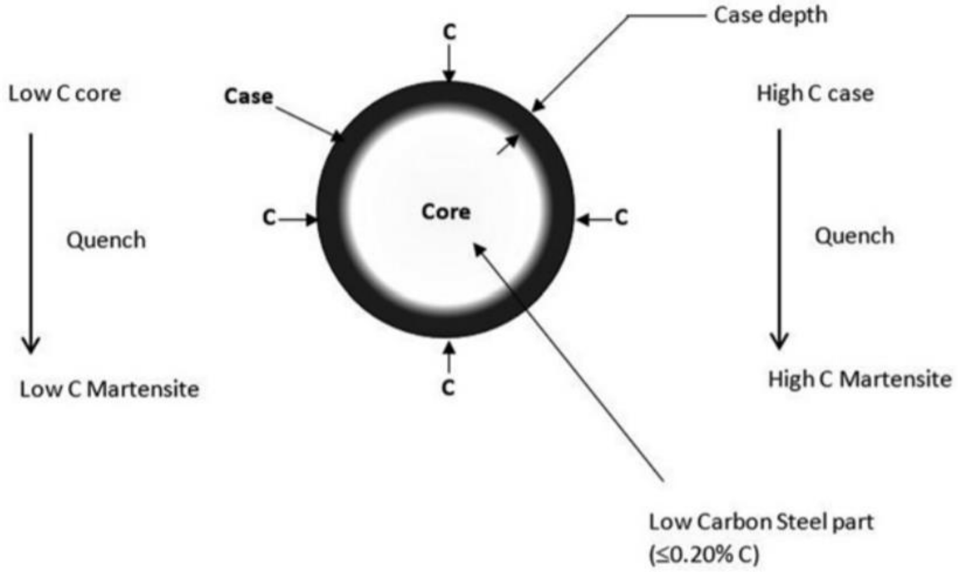


Figure 1. Schematic representation of the carburizing process (Bepari, 2017).

The following Figure 2 shows the types of carburization process. The first three types are the general methods of carburization and they depend on the carburizing medium (Raza et al., 2016). Also, other specialized and modern carburizing techniques such as plasma carburization and vacuum carburization are used nowadays, however, these processes are very expensive (Raza et al., 2016).

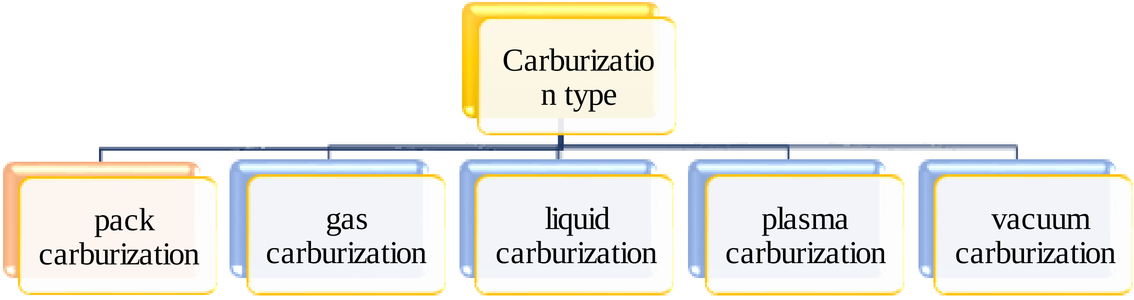


Figure 2. Carburization methods (Raza et al., 2016).

The vast majority of carburized parts are processed by gas carburizing, using natural gas, propane, or butane, to better control for case depth, however, it needs more safety measures (Hosseini and Li, 2016). For liquid carburizing, it is faster than a pack and gas process while it poses a salt disposal problem (Hosseini and Li, 2016). Although the pack carburization process has many drawbacks, it is a dirty and long consuming time process, and it is difficult to control the depth of the case (Abdulrazzaq, 2016).

However, it has many advantages that encourage its use as it is economical and suitable for small machine parts, and does not require a special type of furnace (Bepari, 2017).

The process of solid or pack carburization consists of packing the solid carburizing compound with a specimen of mild steel in a suitable box (Akanji et al., 2015), and heating it slowly in a furnace up to a temperature between 815 °C and 955 °C (Raza et al., 2016). At this temperature range, the dissolving of carbon is higher, and, in terms of mass transfer, the carbon diffuses from the higher concentration to the lower concentration (Bepari, 2017). In the carburization process, when selected the specimen of mild steel usually carbon content is less or equal to 0.20% (Bepari, 2017). This process is not only for steel, but also applies to iron that whose carbon content is less than 0.1% (Verhoeven et al., 2016). Carbon steel is a mixture of iron, carbon, sulfur, phosphorus, silicon, and manganese (Hadi, 2017). And it can be used for structural purposes, in buildings, bridges, and cars (Jabbar and Kadhim, 2020). Figure 3 shows the three types of carbon steel, classified according to the carbon content in it.

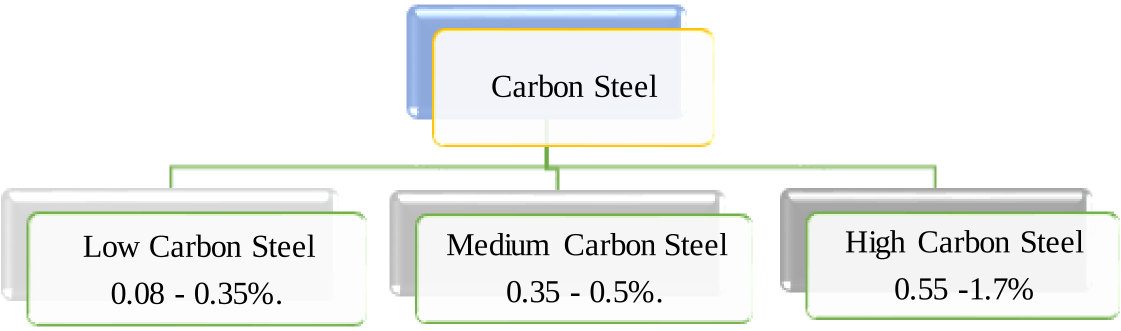


Figure 3. Carbon steel types (Hadi, 2017).

The results of many research papers show that the carburization process improves mechanical properties such as fatigue resistance that will improve when the process takes a longer time (Supriyono and Jamasri, 2017, Shan et al., 2020). Where fatigue failure has four different stages: crack initiation, crack growth, crack propagation, and final rupture (Jabbar and Kadhim, 2020). The fatigue resistance for low carbon steel AISI (1011) increased for specimens subjected to the carburizing process as compared with the same metal which is not treated by this process (Jabbar and Kadhim, 2020). This improvement of fatigue limit was due to the compressive residual stresses and carbide formation at the surface of steel which may be stopping and blocking the crack (Jabbar and Kadhim, 2020).

Traditionally, pack carburizing is carried out using charcoal as a material that provides carbon to the surface of the piece of steel (Raza et al., 2016). The carburizing mixture with smaller particle size means it has a larger surface area that leads to a faster reaction rate and increases tensile strength, hardness, and wear resistance of carburized steel (Adly et al., 2018). Also, the most active carbon source is CB nanoparticles and it can increase the carbon content of low-carbon steel in relatively short times (Raza et al., 2016).

To increase the efficiency of the carburization process, and to reduce the time of it, the best carbon medium should be chosen from several sources (Raza et al., 2016). Carbon sources such as cow bone and coconut shell charcoal where cow bone considered a good source because it has a sufficient carbon element (Rajaguguk and Sumardi, 2019). Also, rice husk can be used as a source of carbon but it has a lower efficiency than traditional charcoal (Llano Martinez et al., 2019). Palm kernel shell and animal bone have good potential to be used as carburizers for mild steel (Madu and Uyaelumuo, 2018). Paraffin wax (sulfur and phosphorous free) and kerosene also are used as carburized material, but paraffin wax has more performance than kerosene (Oreko et al., 2020). However, other sources can be used such as coal, wood charcoal, graphite (Raza et al., 2016), and sugar cane charcoal (Ihom, 2018). When the carburized mixture contains a carbonaceous matter and distilled water this method is called paste carburization, hence, higher mechanical properties of steel can be obtained in less time (Abdullah et al., 2017).

The addition of the energizers such as $BaCO_3$, Na_2CO_3, and $CaCO_3$ to the charcoal and coke leads to accelerating the carburizing reaction also the case depth being higher than case depth without energizer (Supriyono, 2018). Moreover, cow bone can be used as an energizer (Hassan, 2015). Organic waste takes time to degrade in the soil, but the amazing thing is if new and useful uses for it can be found. There is a study that has proven that organic energizers have a better performance than industrial chemical energizers (Aondona and Azoro, 2018). The organic energizers that could be used in the carburization processes are eggshell, cow bone, snail shell, periwinkle shell, banana peel (Aondona and Azoro, 2018), and sea-shell (Akanji et al., 2015).

4. Objectives

The main goals of this project are:

1. Testing the effect of industrial pyrolized tire waste, residual CB, as a carburizer to enhance the hardness and fatigue life for mild steel in the pack carburization process.
2. Testing the effects of carburization temperature and time on the hardness and fatigue life of carburized mild steel.
3. Comparing different types of carbon sources, CB and commercial charcoal, on the mild steel by carburization process.

5. Material and methods

5.1 Materials

The materials of the test specimens used in this study are:

1. The ST 3SP mild carbon steel with %C (0.14-0.22) was sourced from Al-HADDAD INVESTMENT & STEEL CO (Hebron, West Bank) which is manufactured based on Russian standards (GOST_380_2005). The specimen dimensions were 80 mm in length and 12 mm in diameter.
2. The pack carburization container is a box made from heat resistant steel which is St37 (melting point of it is 1600 °C). The dimensions of it as shown in Figure 4 below and the thickness wall is (1) cm. Also, as shown in Figure 5, it has a well-fitting lid to prevent the entry of too much air; however, it should allow the spent gases to escape (Prabhudev, 1988).

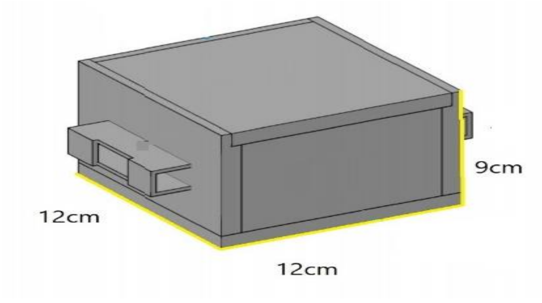


Figure 4. The dimensions of the desined carburization box.

Figure 5. The manufactured carburization container.

3. CB which is produced from the pyrolysis process of car tires, and was sourced from a factory in Jenin. It was sieved with 180 micro mesh.
4. Commercial charcoal was purchased from the Palestinian market.
5. Sodium carbonate (Na_2CO_3), which is available at our university.
6. Water for the quenching process.
7. Sandpaper with grit numbers 320, 400, 500, 600, and 1200 for the grinding process.

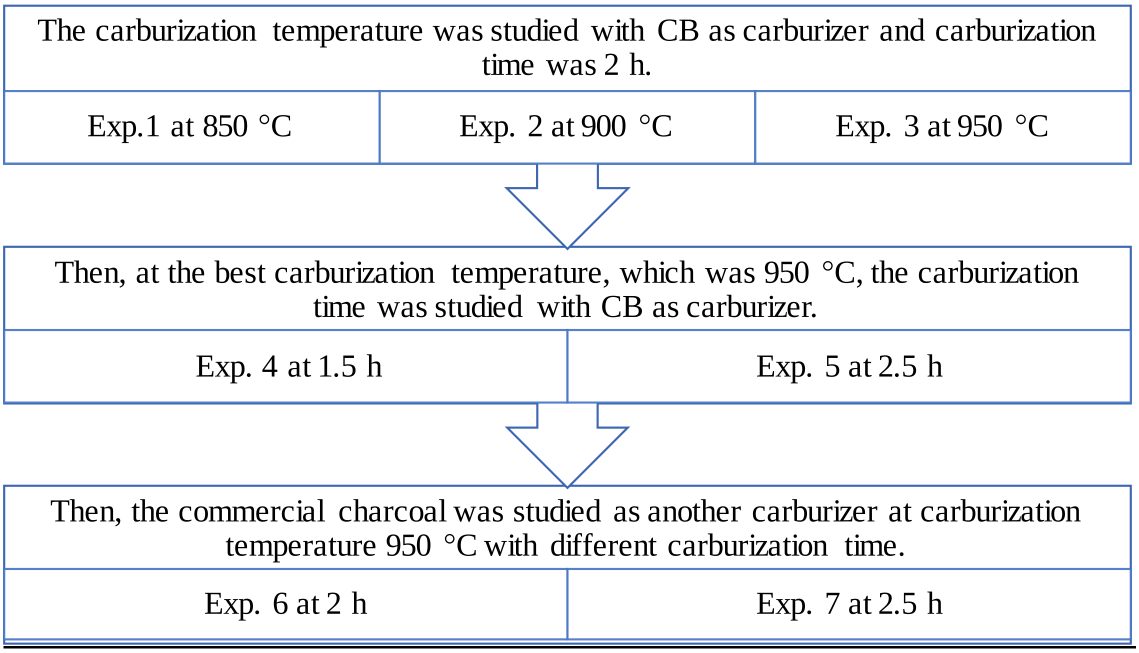
5.2. Methods

5.2.1 The composition of the specimen

The composition of a specimen was tested by a (FOYNDRY MASTER) Optical Emission Spectrometer (OES) in (Al-HADDAD INVESTMENT & STEEL CO) as shown in Figure A in appendices. The test was performed at ambient conditions of temperature and humidity which are 20 °C and 40% relative humidity. OES is a fast, accurate, and reliable method for qualitative and quantitative elemental analysis of metallic samples. The cleaning specimen was mounted on the stand, then electrical energy was applied in the form of a spark was generated between an electrode and specimen. Also, high purity argon was used as a discharge atmosphere for preventing any interaction between the surface of the specimen and the atmosphere. The material was violated from the surface of the specimen resulting in the emission of light, then the light was collected by the spectrometer. In the spectrometer, the incoming light is separated by diffraction grating into an element-specific wavelength, and a corresponding detector measures the intensity of light for each wavelength. The measured intensity is proportional to the offset concentration of the element in the sample (Kumar, 2013).

5.2.2 Pack carburization process

The specimens were charged in a heating resistance metallic box (Figure 7) with a carburizing mixture which was 90% of carbon source and 10% of energizer which is Na_2CO_3. Then the sealed box was placed in an MS8-36 Laboratory burnout furnace (Electrotherm marketing-ShenPaz Technologies, Ramat Gabriel Industrial Park Migdal HaEmek) as shown Figure B in appendices (after heating it to the required carburizing temperature). At the carburizing temperature, the process was held for a required carburizing time. In the first stage, the carburizing temperature was studied with the CB as the source of carbon at a fixed time, which was 2 h. The temperatures were chosen 850, 900, and 950 °C. In the second stage, the carburization process was performed at a fixed temperature, 950 °C, which produced the highest hardness of specimen surface, and the source of carbon was CB, with different carburization times, 1.5 and 2.5 h. In the final stage, another source of carbon, which is commercial charcoal was used as carburization medium in the process at a fixed temperature, which was 950 °C with different times, 2 and 2.5 h. All experiments were performed as shown in the schematic diagram in Figure 6 below.



The carburization temperature was studied with CB as carburizer and carburization time was 2 h.

Exp.1 at 850 °C

Exp. 2 at 900 °C

Exp. 3 at 950 °C

Then, at the best carburization temperature, which was 950 °C, the carburization time was studied with CB as carburizer.

Exp. 4 at 1.5 h

Exp. 5 at 2.5 h

Then, the commercial charcoal was studied as another carburizer at carburization temperature 950 °C with different carburization time.

Exp. 6 at 2 h

Exp. 7 at 2.5 h

Figure 6. All carburization experimental runs were performed in this project. Note: Exp. is the abbreviation of Experiment.

5.2.3 Heat treatment for carburizing specimen

At the end of the carburization process, the furnace was turned off and the box was cooled in the furnace from heating temperature to almost 600 °C, to open the box easily and handle the specimens safely. After that, the specimen was rapidly cooled to room temperature by quenching it horizontally in water according to (Avcı et al., 2009). Then tempering was achieved at a temperature of 550 °C in the furnace for one hour, and it was taken out to be cooled in the air (Aramide et al., 2010).

5.2.4 Hardness test

Hardness can be defined as the resistance measurement of any material to plastic deformation induced by applied forces. The Vickers Hardness test is the most common standard test used to characterize the hardness of the control samples (mild steel before carburization) and carburized samples. The Vickers Hardness formula that is used for calculations is adapted from the textbook Materials science and engineering (Callister and Rethwisch, 2018). The formula reads as follows:

$$\mathrm{HV} = 1.854\ \mathrm{P}/\mathrm{d}_1^{\,2} \tag{1}$$

where:

HV: Vickers hardness.
P: Test load (kgf).
d_1: Average diagonal length (mm).

The following Figure shows the shape of the indentation.

Figure 7. The side view and top view of indentation (Callister and Rethwisch, 2018).

The Vickers Hardness values are multiplied by a correction factor when the surface is cylindrical.

Correction Factors are tabulated in terms of d/D^A as shown in Figure E in appendices, where:

D^A: Diameter of the cylinder in (mm).

d: Mean diagonal of impression in (mm).

But the values of the correction factor were taken after making the extrapolation and the following equation was used as shown in Figure 8.

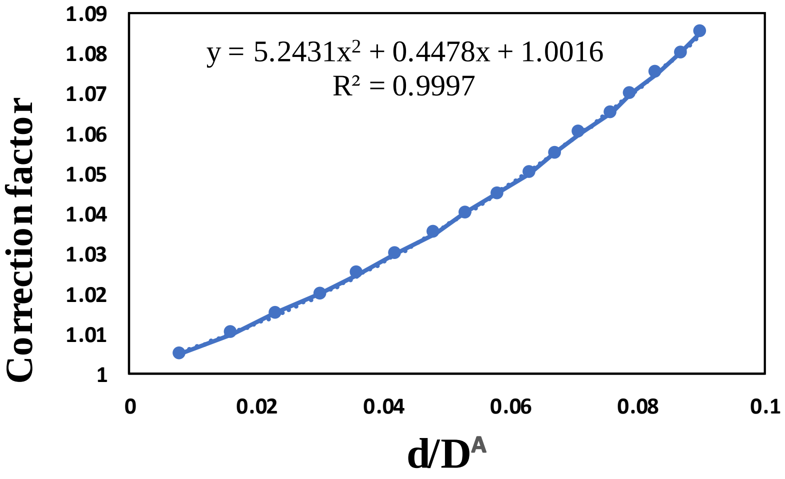


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Figure 8. Correction factor for VHN when the surface is cylindrical.

The specimen was treated before the hardness test by grinding it using a METASERV 2000 TWIN Grinder-Polisher Machine (BUEHLER Company, ILLINOIS USA) as shown in Figure C in appendices.Vickers hardness method was done by (1600-6100) HIGH QUALITY MICRO HARDNESS TESTER Machine (BUEHLER Company, ILLINOIS USA) as shown in Figure D in appendices, which was started by indenting the test material with a diamond indenter, in the form of a right pyramid with a square base and an angle of 136 degrees between opposite faces subjected to a load of 1 kgf (Callister and Rethwisch, 2018). The full load is normally applied for 15 seconds. The two diagonals of the indentation left on the surface of the material after removal of the load are measured using a microscope and their average calculated. The magnification of the lens is 40x for an object lens and 10x for an eyepiece.The area of the sloping surface of the indentation is calculated. The Vickers hardness is the quotient obtained by dividing the kgf load by the square mm area of indentation (Callister and Rethwisch, 2018).

5.2.5 Fatigue test

5.2.5.1 Test specimens

Figure 9 shows the shape and dimensions of the specimens that were prepared according to the standards of the fatigue testing machine used (FARFAN, 2004).

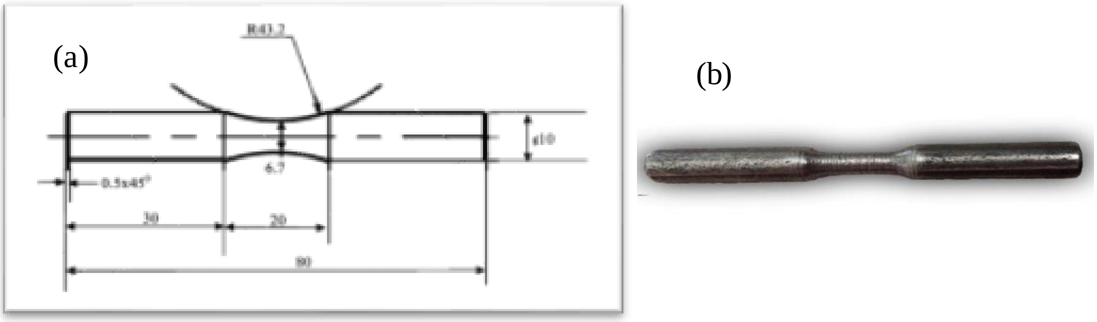


Figure 9. Specimens used in fatigue tests, where the dimensions in mm (FARFAN, 2004): a) - standard dimension specimen b)- real specimen used in experimental works with the same standard dimension.

5.2.5.2 Fatigue testing procedure

The standard high-speed rotating beam test machine was used to conduct fatigue testing. The two specimens from each group were tested individually on this machine (High speed rotating BEAM fatigue test, An-Najah National University, Palestine), each specimen was subjected to a completely reversed stress cycle with a constant force. Each specimen was tested until failure and the corresponding number of stress cycles and the time was recorded. The average number of stress cycles until failure of the two specimens of each group was calculated (Ramahi and Fattah, 2017).

6. Results and Discussion

Three heat treatment processes were performed, namely carburizing, quenching, and tempering. The suitability of using CB as the carburization medium for mild steel was evaluated and it was compared to another carbon source, which is charcoal. Also, the effect of carburization temperature and time on the mechanical properties of mild steel such as hardness and fatigue were studied.

6.1 Percentage of carbon and hardness

6.1.1 Carburization temperature effect

It was noticed that when the temperature is between 850 and 950 °C, the oxygen in the air reacts with the carbon in the carburizer to produce carbon dioxide, and sodium carbonate (energizer) decomposes at this temperature range to produce carbon dioxide

(Hosseini et al., 2013). Then the carbon dioxide further reacts with carbon in the carburizer to produce carbon monoxide (Hosseini and Li, 2016). This carbon monoxide is the carrier of carbon and when comes in contact with austenitic iron, iron carbide is eventually formed, which is dissolved in austenite (Hosseini and Li, 2016). At the initial stage of carburization, the maximum saturation limit of carbon at the surface of the steel is reached (Bepari, 2017). Then carbon begins to diffuse to the interior. For instance, Figure F in appendices shows the equilibrium diagram at any temperature for the reaction in which carbon monoxide on the surface of the steel decomposes into atomic carbon and carbon dioxide.

To address the effect of the carburizing temperature, as previously mentioned, three different temperatures were chosen, which are 850, 900, and 950 °C for 2 h. The following Figure 10 shows the relationship between the carburizing temperature and the percentage of carbon on the surface. It has been observed that the higher the carburization temperature, the greater the percentage of carbon that penetrates the surface of the low carbon steel.

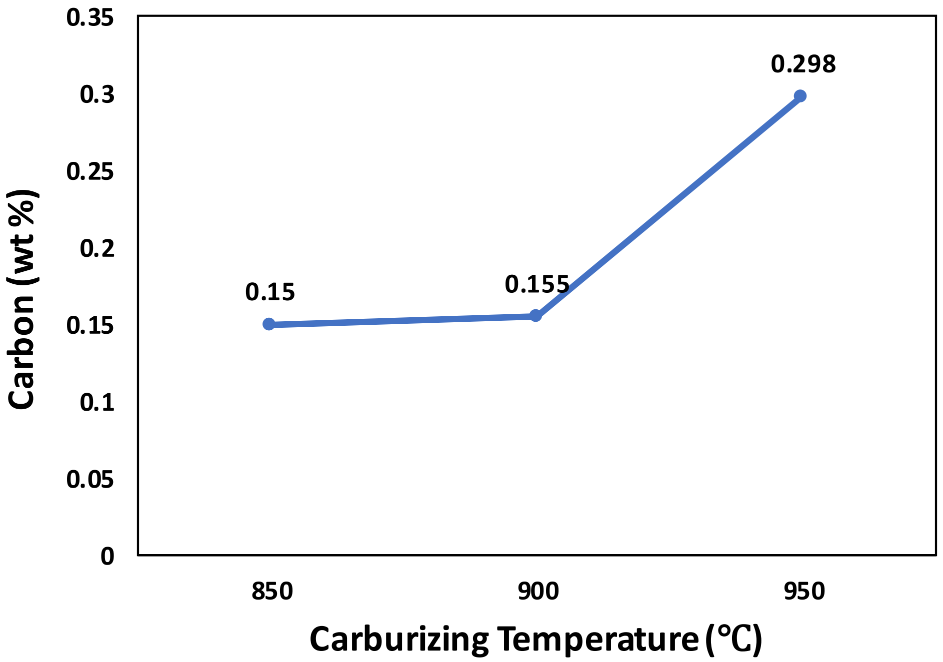


Figure 10. The relationship between the percentage of carbon content and temperature.

At a higher temperature, a greater proportion of the reactants are present with the required activation energy, which increases the rate of the reaction and the temperature dependence of the rate of a chemical reaction can be accurately explained by Arrhenius's equation (H Scott, 2006). Therefore, when the carburizing temperature increases, the vibration of the atoms increases and this helps to increase the diffusion of carbon and depth of penetration (Bepari, 2017).

Besides, the measured hardness values were taken from different positions of the specimen as shown in the following Figure 11, where the average surface hardness values expressed by using the symbol D.

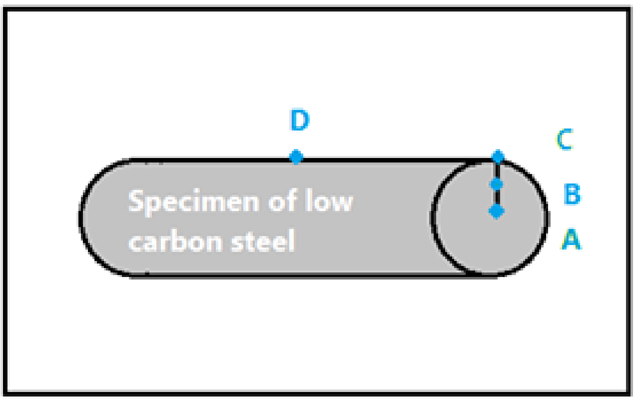
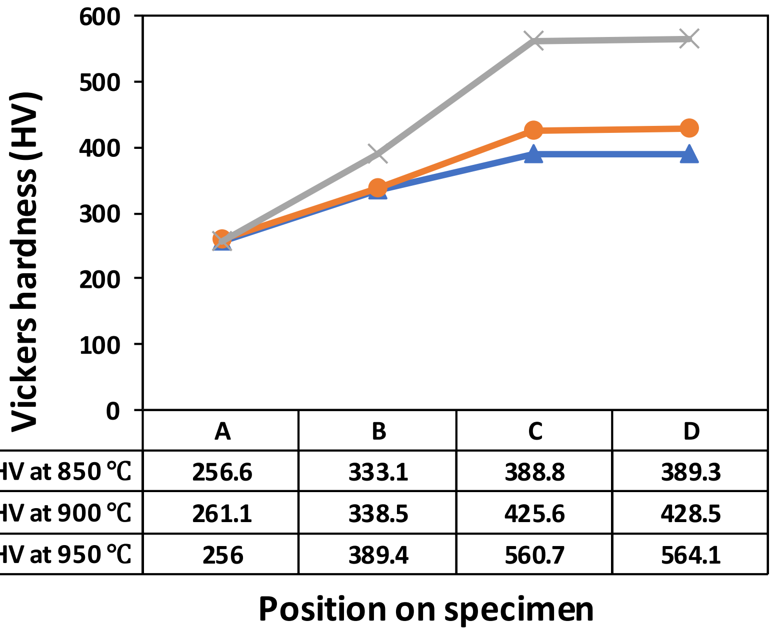


Figure 11. The positions where the hardness values were taken.

A direct positive relationship is observed between the temperature and the hardness value, as seen in Figure 12. In all conditions, hardness decreases with the increase of distance from the surface, because the carbon content of the surface region is more than the core region of steel at the end of carburizing time (Bepari, 2017).



	A	B	C	D
▲ HV at 850 °C	256.6	333.1	388.8	389.3
● HV at 900 °C	261.1	338.5	425.6	428.5
× HV at 950 °C	256	389.4	560.7	564.1

Figure 12. Comparison of Hardness Vickers No. for carburized specimens at temperatures (850, 900, and 950 °C) and for 2 h.

It was observed that there is a relationship between the carbon percentage and the hardness value, for example at a temperature of 950 °C the carbon percentage was 0.298 wt.% (from Figure 10) and it has the highest hardness value. And at 850 °C the carbon percentage equal to 0.150 wt. % (from Figure 10) also it has the lowest Vickers hardness number.

One of the disadvantages of the pack carburization process is that it cannot be obtained the uniform case depth (Bepari, 2017). In other words, this means that the percentage of carbon differs from one position to another in the same specimen, and this was observed because it was reflected in the values of hardness, for example, samples that carburized at 900°C for two hours, and the hardness values were as shown in the following Figure 13. The reason for uneven case depth may be due to an improper packing or uneven heating of the container (Bepari, 2017).

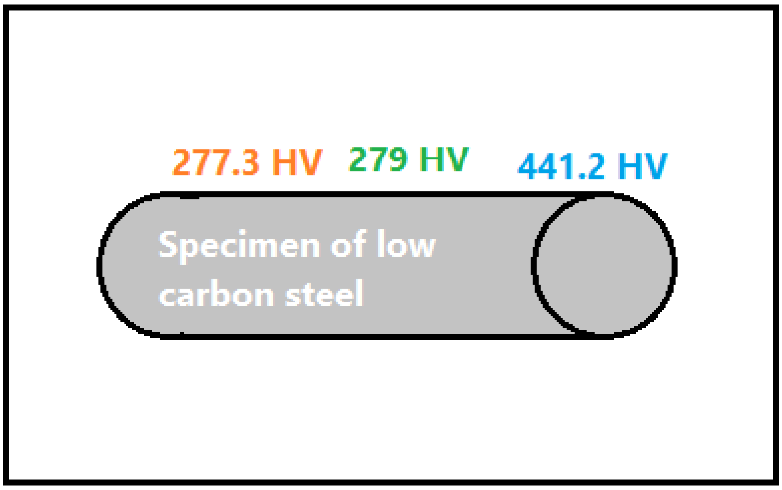


Figure 13. Vickers hardness number at different positions on the surface of carburized steel at 900°C.

The following Table 1 shows many different studies that have been conducted to improve the mechanical properties of carbon steels. From this table, it can be concluded that the higher the temperature at constant time of the carburization process leads to an increase in the hardness value and this result is correct regardless of the carbon source and the type of activator and also whatever the conditions of the heat treatment process that follows the carbonization process. These results are in line to what have been obtained practically in the laboratory, thus, consistent with the literature. This confirms the usefulness of using such cheap residual CB byproduct for the purpose of this study.

Table 1. Effect of temperature on the carburization process.

Type of steel	% C	Source of carbon	Energizer	Comp*	CT* (°C)	Ct* (h)	Heat treatment	Surface hardness (HV)	Reference
AISI 1010 low carbon steel	0.105	Before the carburizing process						103	(Abdulrazzaq, 2016)
		Cook	$CaCO_3$	-	850	2	Quenching in oil	205	
					900			272	
					950			292	
Mild steel	0.28	Before the carburizing process						200	(Adly et al., 2018)
		Coke derived from destructive distillation of low ash, low-sulfur coal Size (1:1.6mm)	-	-	850	2	Quenching it in water then tempering it at 200 °C for 15 min	280	
					900			300	
					950			315	
Mild steel	0.16	Before the carburizing process						-	(Elzanaty, 2014)
		Activated carbon	-	-	850	0.5	Quenching it in engine oil then tempering it at 200 °C for 30 min	551	
					900			640	
					950			694	
Mild steel (ST 3SP)	0.139	Before the carburizing process						260.5	This study
		CB	Na_2CO_3	90% CB 10% Na_2CO_3	850	2	Quenching it in water then tempering it at 550 °C for 1 h	389.3	
					900			428.5	
					950			564.1	

% C*: the carbon content in the untreated sample.
Comp*: composition of the carburization medium.
CT*: the temperature of the carburization process.
Ct*: the duration time of the carburization process.

6.1.2 Effects of the carburization time on the percentage of carbon and hardness

The thickness of the carbon layer increases with increasing time at constant temperature (Bepari, 2017), as shown in Figure G in appendices. To find out the effect of time on the carburization process, the experiment was done at a constant temperature of 950 °C and the time was changed. The results are listed in Table 2.

Table 2. Carburized carbon steel results at 950 °C and at different times.

Time (h)	**1.5**	**2**	**2.5**
Percentage of carbon (wt. %)	0.152	0.298	Not tested*
Vickers Hardness (HV)	415.8	564.1	598.4

Not tested*: The specimen is not tested yet due to coronavirus lockdown.

It can be inferred from the previous table that the carbon percentage and the hardness value increase in proportion to the increase in carburization time. As seen, a higher Vickers hardness number at 2.5 h was achived, this could be attributed to the direct correlation between carbon diffusion (diffusivity) and carburization time (Jabbar and Kadhim, 2020). Harris explained this relationship in an equation in which the square root of time is directly proportional to the depth of the case (Bepari, 2017). So, over time the diffusion of carbon increases. Higher carburization temperature and higher soaking time will result in a large carbide layer and harder case, while core is still tough. Also, the following Table 3 shows the different studies done at a constant temperature but with variable soaking time. All these studies correspond to the study conducted in the laboratory.

Table 3. Effect of time on the carburization process.

Type of steel	% C*	Source of carbon	energizer	Comp*	CT* (°C)	Ct* (h)	Heat treatment	Surface Hardness (HV)	Reference
Low carbon steel	0.17	Before the carburizing process						150	(Supriyono and Jamasri, 2017)
		Charcoal of unused mahogany	Na_2CO_3	80% charcoal 20% Na_2CO_3	930	2	-	310	
						3		340	
						4		345	
AISI 1018 Mild steel	0.18	Before the carburizing process						-	(Akanji et al., 2015)
		Charcoal	Seashell which contains $CaCO_3$ Size: 212µm	90% charcoal 10% Seashell	950	4	Quenching it in water then tempering it at 200°C for 1 h	250	
						6		270	
						8		310	
1.5920 steel	0.21	Before the carburizing process						400	(Elzanaty, 2014)
		Graphite	Na_2CO_3	90% Graphite 10% Na_2CO_3	925	3	Quenching it in oil	720	
						5		748	
						8		738	
						11		648	
Mild steel (ST 3SP)	0.139	Before the carburizing process						260.5	This study
		CB	Na_2CO_3	90% CB 10% Na_2CO_3	950	1.5	Quenching it in water then tempering it at 550°C for 1 h	415.8	
						2		564.1	
						2.5		598.4	

% C*: the carbon content in the untreated sample.
Comp*: composition of the carburization medium.
CT*: the temperature of the carburization process.
Ct*: the duration time of the carburization process.

6.1.3 The influence of the carbon source on the carburization process

The last part of the experiments was done to compare the performance of both carbon black (CB) and charcoal on the carburization process at 950 °C and 2 h.

The results presented in this work show that the use of CB as a carburizing substance in the pack carburizing heat treatment allows being added carbon to the surface of the steel, achieving on this surface an increment of hardness with respect to the hardness presents in the steel without heat treatment. However, the results indicate that the carburizing potential of the CB is lower than that of charcoal. As seen in the following Table 4, the results obtained indicate that the hardness value over 2 h for charcoal was higher than the hardness value of CB. The explanation for this is could be due to the chemical composition of the sources of carburization (Llano Martinez et al., 2019), where the carbon element is the element that most controls the carburization process (Llano Martinez et al., 2019), and with the increase in the carbon content in the carburization source the mechanical properties, are greatly improved compared to the source that has lower carbon content. Nontheless, the other studies in Table 4 show that several sources of carbon used in the pack carburizing of steel indicating different values of surface hardness and affirming our hypothesis.

However, in this study, the CB was used as a new source of carbon for carburizing steel and this source is not better than all the sources used in the carburization process, but it can improve the mechanical properties and thus all the objectives of the project have been achieved.

Table 4. Effect of source of carbon on the carburization process.

Type of steel	% C*	Source of carbon	energizer	Comp*	CT* (°C)	Ct* (h)	Heat treatment	Surface Hardness	Reference
Low carbon steel	0.106	Before carburizing process						57.15HRC	(Miswanto, Rajaguguk and Sumardi, 2019)
		Cow bone	-	-	950	3		61.43HRC	
		Coconut shell						64.3HRC	
Plain carbon steel 1024	0.24	Without	$BaCO_3$	80% source of carbon 20% $BaCO_3$	900-950	3	-	140HV	(Raza et al., 2016)
		Nano particles Carbon black						275HV	
		Nano particles Charcoal						148HV	
SAE 1020 mild steel	0.199	Before carburizing process						171HB	(Llano Martinez et al., 2019)
		Charcoal	$CaCO_3$	60% source of carbon 40% $CaCO_3$	950	7	-	254HB	
		Husk rice						233HB	
Mild steel (ST 3SP)	0.139	Before carburizing process						260.5HV	This study
		CB	Na_2CO_3	90% source of carbon 10% Na_2CO_3	950	2	Quenching it in water then Tempering it at 550°C for 1 h	564.1HV	
		Charcoal						820HV	

6.2 Fatigue strength.

6.2.1 Effects of temperature on the number of cycles and fatigue resisance

Fatigue strength is an important mechanical property (Jabbar and Kadhim, 2020), and it can be determined by multiplying the fatigue strength of the material with the reduction factors (Supriyono and Jamasri, 2017). An example of this factors, surface factor, geometry factor, temperature factor, etc. The surface factor is considered the most common one. However, the failure of fatigue starts on the metal surface (Jabbar and Kadhim, 2020) and propagates across the volume of the component (Supriyono and Jamasri, 2017). Fatigue failure has four different stages: crack initiation, crack growth, crack propagation, and final rupture. Most surface treatments produce compressive stresses in the metal surface, which reduce the probability of crack initiation and its expansion at the interface between the surface and core, thus increasing resistance to fatigue (Jabbar and Kadhim, 2020).

It is obvious from the results in Table 5 that there is a relationship between the number of stress cycles and the hardness at the same time (2 h), when the surface hardness increased the value of the number of stress cycles decreased, this is true only for the specimens at 950 °C and 850 °C both at 2 h. However, for the specimens at 900 °C at 2 h, it showed a rise in their number of cycles, knowing that its hardness higher than the 850 °C at 2 h and lower than 950 °C at 2 h. So, there is a need for more study and do more experiments. But this improvement of fatigue resistance for specimen at 900 °C was due to maybe the compressive residual stresses and carbides formation at the surface of steel which may be stopping and blocking the crack, besides, the increase in the case depth, carburizing layer, or carbon layer plays a role in increasing the fatigue resistance. In terms of the microstructure of the specimen, refinement of the austenitic grain size developed in the carburizing layers would improve fatigue resistance (Loganathan et al., 2011) and (Abdulrazzaq, 2016). It is also the same relation between the number of stress cycles and the temperature as shown in the table below.

Table 5. Values of the number of stress cycles and the hardness at time 2 h and at different temperatures.

Temperature	**850**	**900**	**950**
Vickers Hardness (HV)	389.3	428.5	564.1
Number of cycles	36767.5	178496.5	25603

So, the above results indicate that the specimens with the lowest number of cycles have the lowest fatigue resistance (Sujita et al., 2018).

6.2.2 Effects of time and the carbon source on number of cycles and fatigue resistance

The holding time during the carburizing process also influences the fatigue resistance of the material. The longer the holding time will have the higher number of cycles and the fatigue resistance (Supriyono and Jamasri, 2017). Where all studies shown in Table 6 below showed that the number of cycles increased with increasing the time of the carburization process in the same value of fatigue strength and temperature, which leads to an increase in fatigue resistance, and this result is correct regardless of the carbon source and the type of activator and also whatever the conditions of the heat treatment process that follows the carbonization process. These results correspond to the results obtained in the laboratory, where at 2 h the number of cycles was 25603, while at 2.5 h the cycles were 35331 at the same temperature 950. But at the holding time of 1.5 h, the number of cycles increased more than 2 h and 2.5 h to reach 124467cycles. So, there is a need for more study and do more experiments to explain these results, due to their differs from what has been reported in the literature.

Table 6: Effect of time on the fatigue resistance by calculating the number of cycles.

Type of steel	% C*	Source of carbon	energizer	Comp*	CT* (°C)	Ct* (h)	Heat treatment	σ* (MPa)	Fatigue (number of cycles)	Reference
Low carbon steel	0.17	Before carburizing process						160	60000	(Supriyono and Jamasri, 2017)
		Charcoal of unused mahogany	Na_2CO_3	80% charcoal 20% Na_2CO_3	930	2	-		90000	
						3			95000	
						4			100000	
Low carbon steel AISI (1011)	0.09	Without						-	1210000	(Jabbar and Kadhim, 2020)
		Charcoal	$CaCO_3$ $BaCO_3$ Na_2CO_3	80% charcoal 10 $CaCO_3$ 9 % $BaCO_3$ 1% Na_2CO_3	950	5	-		2500000	
Mild steel (ST 3SP)	0.139	Before carburizing process						642	3255	This study
		CB	Na_2CO_3	90% source of carbon 10% Na_2CO_3	950	1.5	Quenching it in water then tempering it at 550°C for 1 h		124467	
						2			25603	
						2.5			35331	
		Charcoal				2			656	
						2.5			814	

% C*: the carbon content in the untreated sample.
Comp*: composition of the carburization medium.
CT*: the temperature of the carburization process.
Ct*: the duration time of the carburization process.
σ*: bending stress (MPa)

The last row in Table 6 above compares the performance of both CB and charcoal on the fatigue resistance at 950 °C and at two different times which are 2 h and 2.5 h, where it shows that the number of cycles for charcoal at 2h and 2.5 h was 656 and 814, respectively. As their number of cycles is less than the untreated specimen, so, there is a need for more study and do more experiments to explain these results. Where the number of its cycles decreased compering to CB due to the higher hardness, so charcoal has a lower fatigue resistance than CB (Sujita et al., 2018).

Carburizing process does not harden the steel, however, it increases the carbon content to some predetermined depth below the surface to a sufficient level to allow subsequent quench hardening (Maisuradze and Kuklina, 2018). If the steel is allowed to cool slowly, the carbon will separate out of the ferrite as the cubic-structure will change from face-centered back to body-centered (Prabhudev, 1988). The cementite will reform within the ferrite, and the carbon steel will have the same properties that it did before it was heated, however, when the steel is rapidly cooled, or quenched, in a quenching medium (such as water or engine oil) the carbon does not exit the cubic structure of the ferrite and it becomes bonded with the structure (Prabhudev, 1988). This leads to the formation of martensite, which is the microstructure that produces the most sought after mechanical properties in steel fasteners (Bepari, 2017). So, the carburized specimen was quenched in water. The steel which has been quenched from austenitizing temperatures requires tempering before it can be placed into service due to the brittleness property in martensite (Hassan, 2015). Tempering is a process of heating the carbon steel to a specific temperature below that transformation line and the carbon steel is allowed to cool slowly (Aramide et al., 2010). The slow cooling process will increase the ductility and decrease the hardness of a specified level of the crystal structure (Aramide et al., 2010). So, after quenching the carburized specimen in water, it was tempered at 550 °C for 1 h, then it was taken out to be cooled in the air.

7. Conclusions and recommendations

This study presents experiments of the carburizing process at different temperatures, times, and sources of carbon to show their effect on the hardness and fatigue life on the mild steel ST 3SP specimen with %C (0.14 – 0.22). The following conclusions can be drawn:

1. It was found that there is a direct relation between the temperature and the hardness value; the hardness could be increased up to 564 HV by increasing the temperature to 950 °C. This improvement in the hardness is due to the increase in the content and the case depth of carbon on the surface.
2. The relationship between the carbon percentage and the hardness value was also observed where at a temperature of 950 °C the carbon percentage was 0.298 wt.% and it has the highest hardness value.
3. The carbon wt% and the hardness value increase proportionally, when the time of the carburization process was increased, the maximum hardness was around 598 HV at the time of 2.5 h.
4. Charcoal performs better than carbon black (CB) in hardness at 2 h and 2.5 h, but the hardness for 2.5 h was less than 2 h in charcoal, this low hardness mainly due to a higher case depth, the presence of retained austenite.
5. There is a relationship between the number of stress cycles and the hardness at the same time (2 h), so as the hardness was increased the value of the number of stress cycles was decreased. The maximum number of stress cycles became around 178500 cycles at 900 °C.
6. It was found that the specimens with the lowest number of cycles have the lowest fatigue strength.
7. The holding time during the carburizing process influences the fatigue strength of the material by increasing the number of cycles and so the fatigue strength. Where at 2 h the number of cycles was 25603, while at 2.5 h the cycles were 35331 at the same temperature 950 °C.
8. Charcoal has a lower fatigue strength than CB, where its number of cycles were at 2 h and 2.5 h are 656 and 814, respectively. This decrease in the number of cycles comparing to CB was due to the higher in their hardness, But there is a need for more study and do more experiments to explain these results.

Nevertheless, here we recommend the following for any future work on this project:

1. The composition test shall be completed for not tested carburized specimens.
2. The pack carburizing experiment (2) (at 900 °C for 2 h by CB as a carburizer) and experiment (4) (at 950 °C for 1.5 h by CB as a carburizer), also the experiments for the charcoal at (2, 2.5) h shall be repeated for fatigue specimens, to repeat the fatigue test for them, because the fatigue results for them have a deviation from the fatigue results for untreated and other specimens.
3. Conduct experimental tests Such as TGA (Thermogravimetric Analysis) and FTIR (Fourier Transform Infrared) to better explain the results and to know the amount of carbon and the organic content in the CB and Charcoal and that is because it plays a very large role in the reaction of the carburization and to know the functional group found on the CB and charcoal by FTIR.
4. Conduct the microstructure analysis of the specimen before and after carburizing and quenching.
5. To conform with the Russian standards for ST 3SP, as shown in Figure I in appindeces, it is necessary to conduct and measure the other mechanical properties such as tensile strength, yield strength, toughness, and others.
6. The feasibility study for CB can be done as a carburizer for steels in place of charcoal.
7. Conduct experiments by changing the quenching process parameters such as quenching media and quenching orientation.
8. Conduct experiments by changing the tempering process parameters such as tempering temperature and holding time.

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Appendices

These figures that were used in this study.

The (FOUNDRY MASTER) optical emission spectrometer (OES) in (Al-HADDAD INVESTMENT & STEEL CO) used for composition testing of specimens as shown in Figure A.

Figure 14. FOUNDRY MASTER OES.

Figure B shows the MS8-36 Laboratory burnout furnace (Electrotherm marketing-ShenPaz Technologies, Ramat Gabriel Industrial Park Migdal HaEmek) that used for pack carburization process.

Figure 15. MS8-36 Laboratory burnout furnace.

Figure C shows the METASERV 2000 TWIN Grinder-Polisher Machine (BUEHLER Company, ILLINOIS USA) that used for grinding specimens by sand papers.

Figure 16. METASERV 2000 TWIN Grinder-Polisher Machine.

Figure D shows the (1600-6100) HIGH QUALITY MICRO HARDNESS TESTER Machine (BUEHLER Company, ILLINOIS USA) for Vickers Hradness test.

Figure 17. HIGH QUALITY MICRO HARDNESS TESTER Machine.

The correction factors for using the Vickers Hardness test are shown in Figure E, but the correction factor values were used in this study for the Vickers Hardness test are not found, since they were taken after the extrapolation was performed.

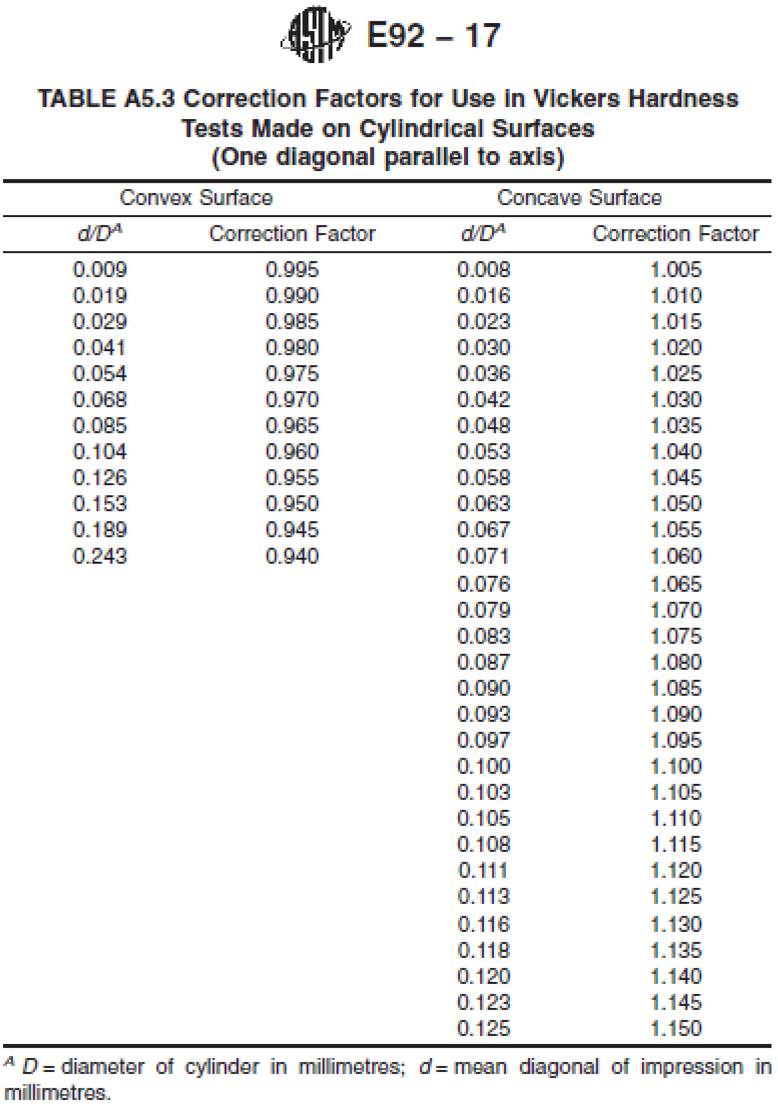
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TABLE A5.3 Correction Factors for Use in Vickers Hardness Tests Made on Cylindrical Surfaces (One diagonal parallel to axis)

Convex Surface		Concave Surface	
d/D[A]	Correction Factor	d/D[A]	Correction Factor
0.009	0.995	0.008	1.005
0.019	0.990	0.016	1.010
0.029	0.985	0.023	1.015
0.041	0.980	0.030	1.020
0.054	0.975	0.036	1.025
0.068	0.970	0.042	1.030
0.085	0.965	0.048	1.035
0.104	0.960	0.053	1.040
0.126	0.955	0.058	1.045
0.153	0.950	0.063	1.050
0.189	0.945	0.067	1.055
0.243	0.940	0.071	1.060
		0.076	1.065
		0.079	1.070
		0.083	1.075
		0.087	1.080
		0.090	1.085
		0.093	1.090
		0.097	1.095
		0.100	1.100
		0.103	1.105
		0.105	1.110
		0.108	1.115
		0.111	1.120
		0.113	1.125
		0.116	1.130
		0.118	1.135
		0.120	1.140
		0.123	1.145
		0.125	1.150

[A] D = diameter of cylinder in millimetres; d = mean diagonal of impression in millimetres.

Figure 18. Correction Factors for use Vickers Hardness test (ASTM E92-17).

According to (Hosseini et al., 2013), if the ratio of carbon monoxide to carbon dioxide at a constant temperature is more than the equilibrium ratio of carbon monoxide to carbon dioxide, this reaction goes in the right direction, and the carburizing phenomenon happens. But when the ratio of carbon monoxide to carbon dioxide at a constant temperature is less than the equilibrium ratio of carbon monoxide to carbon dioxide, the reaction goes in the left direction, and the decarburizing phenomenon happens. Figure F shows that.

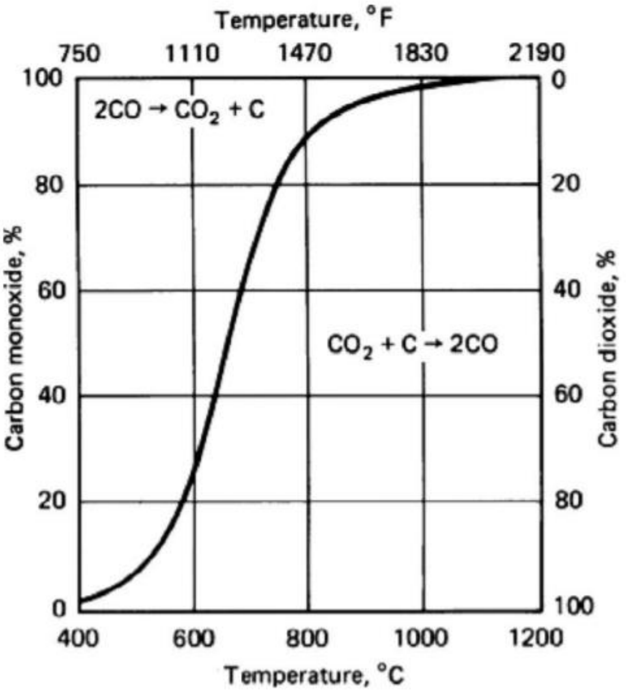


Figure 19. Equilibrium pressure of CO and CO2 for 2CO ↔ CO2 + C $_{\text{atom}}$ reaction (Hosseini et al., 2013).

Figure G shows that the thickness of carbon layer increasing with increasing time (Bepari, 2017)

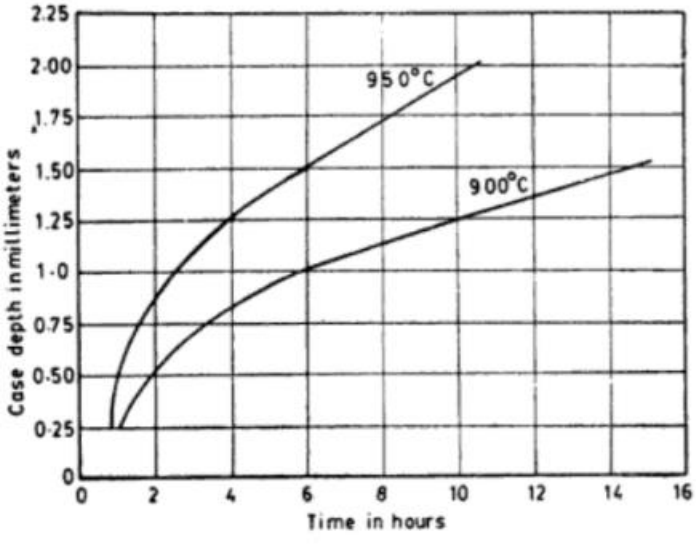
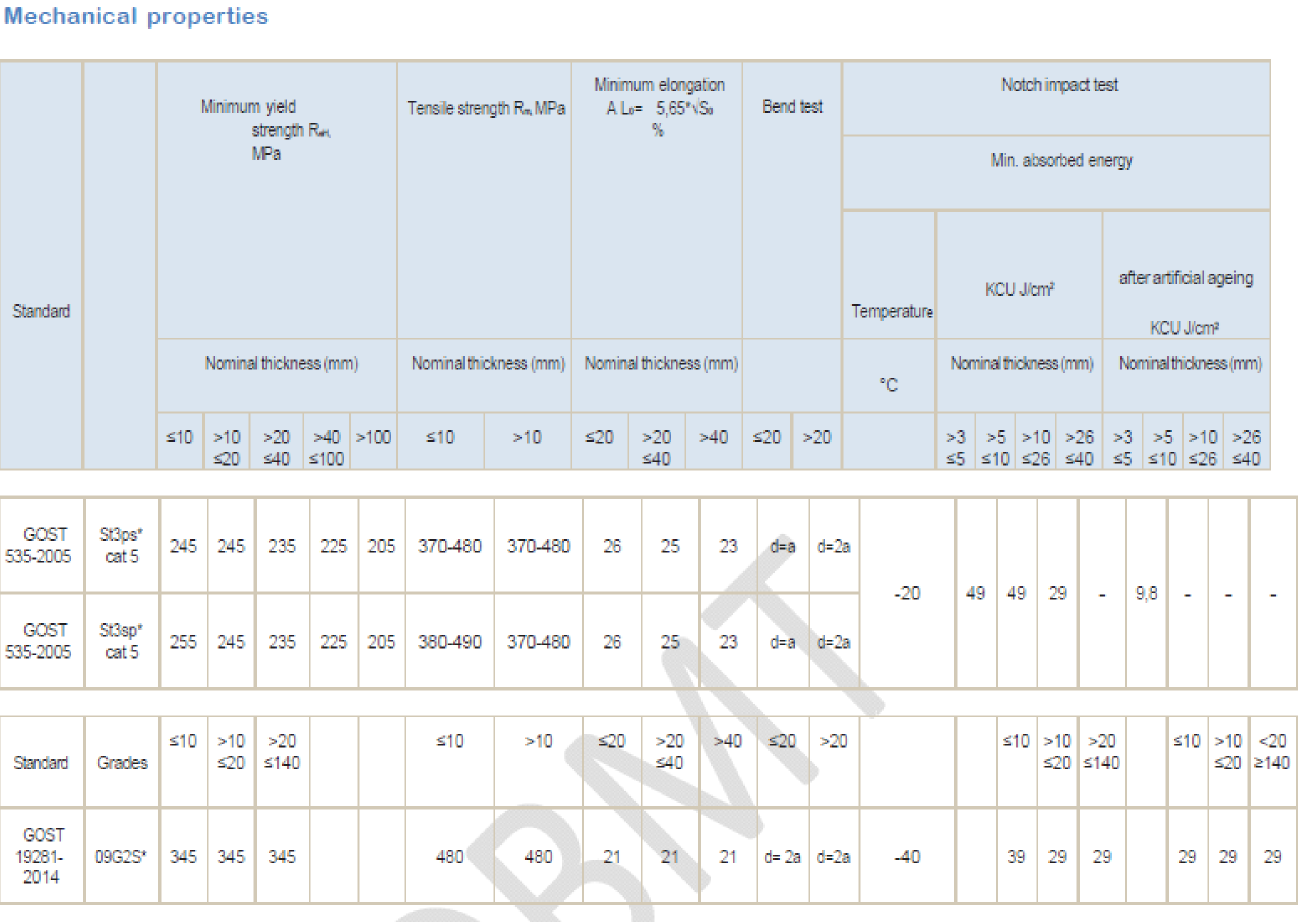


Figure 20. Case depth penetration curve for pack carburizing (Prabhudev, 1988).

Figure I shows the Russian standards for mechanical properties of ST 3SP (mild steel) has been used in this study.



Mechanical properties

Standard		Minimum yield strength R_{eH}, MPa					Tensile strength R_m, MPa		Minimum elongation A L_0= 5,65*$\sqrt{S_0}$ %			Bend test		Notch impact test Min. absorbed energy Temperature	KCU J/cm²				after artificial ageing KCU J/cm²			
		Nominal thickness (mm)					Nominal thickness (mm)		Nominal thickness (mm)					°C	Nominal thickness (mm)				Nominal thickness (mm)			
		≤10	>10 ≤20	>20 ≤40	>40 ≤100	>100	≤10	>10	≤20	>20 ≤40	>40	≤20	>20		>3 ≤5	>5 ≤10	>10 ≤26	>26 ≤40	>3 ≤5	>5 ≤10	>10 ≤26	>26 ≤40
GOST 535-2005	St3ps* cat 5	245	245	235	225	205	370-480	370-480	26	25	23	d=a	d=2a	-20	49	49	29	-	9,8	-	-	-
GOST 535-2005	St3sp* cat 5	255	245	235	225	205	380-490	370-480	26	25	23	d=a	d=2a									
Standard	Grades	≤10	>10 ≤20	>20 ≤140			≤10	>10	≤20	>20 ≤40	>40	≤20	>20			≤10	>10 ≤20	>20 ≤140		≤10	>10 ≤20	<20 ≥140
GOST 19281-2014	09G2S*	345	345	345			480	480	21	21	21	d= 2a	d=2a	-40		39	29	29		29	29	29

Figure I. Russian standards for mechanical properties of ST 3SP (GOST _535_2005).