EPI International Journal of Engineering
Volume 6, Number 1, February 2023, pp. 18-23
DOI: 10.25042/epi-ije.022023.03

*p*ISSN 2615-5109
*e*ISSN 2621-0541

Increased Hardness Value due to the Diffusion of Low-Temperature Carburizing Process

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Abstract

This research to determine the effect of the tensile load on the carburizing process and the heating temperature lower than the normal carburizing temperature on the hardness of carbon steel with a percentage of 80% buffalo bone charcoal and 20% $BaCO_3$, 20 mesh grain size, 4 hours holding time, and slow cooling with a tensile load of $1/4\sigma_p$, $1/2\sigma_p$, $3/4\sigma_p$, and σ_p (proportional stress). The heating temperatures below normal carburizing temperatures, namely 600^0C, 650^0C, 700^0C, and 750^0C while being pulled by $1/4\sigma_p$, $1/2\sigma_p$, $3/4\sigma p$, and σ_p. After reaching the heating temperature, the material is held in the furnace for 4 hours and cooled slowly. After the material is cold, mechanical testing is carried out with Vickers microhardness. Hardness value at a temperature of 600^0C is 103.93 HRB, at a temperature of 650^0C is 104.33 HRB, at a temperature of 700^0C is 104.80 HRB, and at a temperature of 750^0C is 106.60 HRB, while the process of pack carburizing without tensile at a heating temperature of 800^0C is 105.2 HRB. This proves that the application of loads at lower heating temperatures during the heating process can exceed the hardness value without tensile loads at higher temperatures of 800^0 C.

Keywords: Carburizing; low-temperature; buffalo bone charcoal; hardness; tensile load

1. Introduction

This surface-hardening process is greatly influenced by the amount of carbon content contained in the steel. Carbon steel is a mixture of iron and carbon plus elements sulfur (S), Posfor (P), silicon (Si), and manganese (Mn). One way to do surface hardening is by grounding process with solid carbon media or pack carburizing. In the carburizing process of low carbon steel using a mixture of coconut shell charcoal and $BaCO_3$, the heating temperature is 980^0C and the holding time is 4 hours and continued with the quenching process. From the results of metallography on carburized material, on the outside can be seen the microstructure of martensite and the middle of ferrite-pearlite. This indicates the occurrence of surface hardening with the addition of carbon elements on the surface of the test material [1].

Heat treatment of carbon steel is based on thermochemical principles with a diffusion system, which is a way to change the surface properties of the substrate, then additional materials are needed from the outside and the additives will diffuse to the surface of the substrate. Heat treatment of steel is also based on the principle of physical metallurgy relating to processes, properties, and microstructure. In the heat treatment process, the whole process uses heat to change the steel structure. Changing the surface properties of steel can be done by changing the structure and shape of the surface with thermomechanical treatment [2].

Another research by improving the quality of metals, especially their hardness by the carburization process. According to research [3] which examines the treatment of pack carburizing on low carbon steel as an alternative material for cutting knives where the process of pack carburizing is in a kitchen room is heated gradually, the first stage is 200ºC for 1 hour, the second stage is 500ºC for 1 hour and 700ºC for 1 hour, finally at a carburizing temperature of 900ºC for 1 hour. Then cooling is done slowly, where the kitchen is turned off and waited until it drops to a temperature of 350ºC. After reaching that temperature, the kitchen door is opened to remove the carburizing box. Outside the furnace, the lid of the carburizing box is opened, and all specimens are removed to be cooled openly.

Previous research has also shown that the use of local media in this case cow bone ($CaCO_3$) can be used as an alternative to the catalyst $BaCO_3$ (barium carbonate) in the solid carburizing process. With the greater number of catalyst granules, the fastest carbon absorption rate occurs in the process with a holding time of 15 minutes with a composition of 1 kg of a mixture consisting of 70% carbon (nani wood charcoal) and 30% cow bone ($CaCO_3$) with a grain size of 5 mm which is 2.89 HRC. Then followed by

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the magnitude of catalyst granules of 3 mm and 1 mm with values of 155.19 HRC and 154.18 HRC, where the largest increase in the average hardness rate occurred in the magnitude of 5 mm catalyst granules of 155.90 HRC or 35.90% increased from the initial hardness value [4].

Other research results showed that carburizer with goat bone charcoal provided the highest surface hardness (556.37 HV) followed by bamboo charcoal (532.01 HV), coconut frond charcoal (363.41 HV), and duck bone charcoal (340.41 HV). Meanwhile, in terms of depth of hardness, carburizing with bamboo charcoal provides the highest depth effectiveness of up to 2.6 mm followed by carburizing with goat bone charcoal, duck bone charcoal, and coconut frond charcoal with hardness depths of 1.4 mm, 1.2 mm, and 1 mm respectively [5].

Heat tensile tests were performed at temperature variations of 850ºC, 900ºC, and 950ºC and strain rates of 0.01 and 1/sec. The results of the heat tensile test show that the higher the temperature, the lower the maximum tensile stress and flow stress of S48C steel. The highest maximum stress drop occurred at 950ºC at 85% of room temperature conditions, while the highest flow stress drop occurred at a 950ºC test temperature of 31% compared to 850^{0}C, strain (ε) 0.23 μ speed/strain rate ($\acute{\varepsilon}$) 1/sec by 27% compared to temperature with the same strain but $\acute{\varepsilon}$ 0.01/sec. An increase in strain rate from 0.01/sec to 1/sec in the temperature range of 850ºC-950ºC will increase the flow stress by 46-53% [6].

Based on this research, the author wants to develop a pack carburizing process at temperatures lower than normal carburizing temperatures. This method can be done by tensile hot conditions to the proportional limit of carbon steel material without the deformation of carbon steel. With the aim of obtaining the same hardness as the pack carburizing process but a smaller temperature, it can be done external force with a load while the pack carburizing process is carried out. Metal in hot conditions when given a load will facilitate strain so that the distance between atoms (interspace atoms) of carbon steel will be greater, making it easier for carbon to diffuse into steel. Where the increase in hardness occurs as the temperature rises, the atoms vibrate with greater energy, and a small number of atoms will move within the lattice. When atoms fill the void, a new hole or void occurs. This new void can be filled by other atoms coming from around the material. As a final result, it can be said that atoms carry out random motion in crystals. The mechanism of random motion can be applied to carbon atoms moving between iron atoms, from one insert position to the next [7]–[9].

With this study to determine the hardness of the material after a carburizing process with buffalo bone charcoal which is given a withdrawal load by not exceeding its proportional limit

2. Literature Review and Problem Statement

Chemical heat treatment in steel is the process of heating steel by adding certain substances when heating, then cooling. This chemical heat treatment can be in the form of (1) carburizing, (2) nitriding, (3) cyaniding or carbonitriding, and (4) diffusion coating. Carburizing is the process of coating the surface of steel with carbon by heating steel at a temperature of 750ºC-950ºC, where the process of heating and cooling the metal in a solid state changes the physical and mechanical properties of the metal [10].

This movement will be followed by the movement of other adjacent atoms until stable conditions occur and this event generally takes place through two mechanisms, namely interstitial and vacancy [9].

The structure of ferrite (iron α) and austenite (iron γ) has the ability to accommodate insertor atoms such as carbon atoms to form solid solutions. Because the size of the carbon atom is relatively small compared to the iron atom, it allows the carbon atom to enter the lattice of α iron and iron γ as interstitial soluble atoms. Metal alloying elements such as manganese, nickel and chrome have relatively large atoms so that when they enter iron will form a solid solution substitution therefore, the comparison of the size of carbon atoms with the size of the available gaps shows that some distortion will occur when carbon atoms enter the iron lattice [11].

This movement will be followed by the movement of other adjacent atoms until stable conditions occur [9], [11], [12]. Diffusion in metals can be affected by many factors, including temperature, pressure, material composition, grain size, and type of crystal defect. For example, at high temperatures, metal atoms can move more easily and the diffusion process can occur faster, while at low temperatures, the diffusion process can be slower. In materials engineering, understanding and controlling diffusion in metals is essential to achieve the desired properties and performance in practical applications [12]–[14].

Temperature has the most influence on the coefficient and rate of diffusion. For example, for self-diffusion Fe in -Fe, the diffusion coefficient increases approximately sixfold (from 3.0 to 1.8 in temperature rise from 500ºC to 900ºC (Table 1). Temperature has the most influence on the coefficient and rate of diffusion [8]. The temperature dependence can be seen in Table 1 [15].

Bone charcoal (carbo animalis) is a porous, black, grained material produced from burning animal bones. The composition varies depending on how it is made, but consists mostly of tricalcium phosphate (or hydroxyapatite) 57–80%, calcium carbonate 6–10% and carbon 7–10% [16]. It is mainly used for filtration and decolorization.

Buffalo has a very important role for the livestock economy as a producer of milk, meat, and labor [17], 2015). Buffalo is also the most important animal in the social life of Torajan society, because it is a slaughter animal at the ceremony of the dead or bereavement [18]. Based on data from research [19], the number of buffalo slaughtered for mourning ceremonies is around 13.000 heads per year. This very large number of buffalo will certainly cause a lot of bone waste. In response to this, an alternative is used, namely as an adsorbent. Adsorbent is a

Table 1. Tabulation of diffusion data

Diffusing Species	Host Metal	D_o(m^2/s)	Activation Energy Q_d		Calculated Values	
			kJ/mol	eV/atom	T(°C)	D(m^2/s)
C	α-Fe	6.2×10^{-7}	80	0.83	500	2.4×10^{-12}

substance that has binding properties on the surface and this property is very prominent in porous solids [20], the organic content of buffalo bones is 35%. The carbon content in bones is quite a lot so that it is very possible to be used as raw material for making activated charcoal. Activated charcoal is an amorphous compound that can be produced from materials containing carbon or from charcoal that is specially treated to obtain a wider surface. The surface area of activated charcoal ranges from 400-800 m^2/gram with pore sizes between 5-10 A. Bone charcoal has a high absorbency because bone charcoal has a large amount of pores. To utilize buffalo bones as a source of carbon, it will be processed into charcoal then as a carburizing medium in the carburizing process.

From the above, the author wants to develop a pack carburizing process at temperatures lower than normal carburizing temperatures by tensile hot conditions to the proportional limit of carbon steel material without the deformation of carbon steel. This pack carburizing method is carried out at temperatures lower than normal carburizing temperatures, namely 600^{0}C, 650^{0}C, 700^{0}C, and 750^{o}C with tensile loads of $1/4\sigma_p$ $1/2\sigma_p$ $3/4\sigma_p$, and σ_p and buffalo bone charcoal as an energizer source and $BaCO_3$ as a catalyst with a percentage of 80%ATK + 20%$BaCO_3$, holding time 4 hours, mesh 20, and slow cooling.

3. Aims and Objectives of the Study

This study aims to determine the effect of tensile with a tensile load not exceeding its proportional stress in the solid carburizing process and heating temperature lower than the normal carburizing temperature on the hardness of carbon steel. To achieve this goal, a pack carburizing process was carried out with a percentage of 80% buffalo bone charcoal and 20%$BaCO_3$, 20 mesh grain size, 4 hours holding time, and slow cooling with a tensile load of $1/4\sigma_p$, $1/2\sigma_p$, $3/4\sigma_p$, and σ_p (proportional stress). Each treatment at each different temperature was tested with 3 specimens per temperature and the hardness value of each specimen was averaged to obtain hardness values at temperatures of 600^{o}C, 650^{o}C, 700^{o}C, and 750^{o}C and each withdrawal load. Hardness testing using the micro hardness method.

4. Research Materials and Methods

4. 1. Material and tools

a. Materials

The materials used are:

1. Carbon steel (Fig. 1).
2. Buffalo bone charcoal (Fig. 2).

Figure 1. Carbon steel shapes for specimens

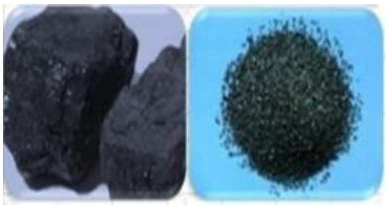

Figure 2. Buffalo bone charcoal

Figure 3. Barium Carbonate ($BaCO_3$)

3. Barium Carbonate ($BaCO_3$) (Fig. 3)

b. Equipment

The equipment used in this study are:

1. Furnance (Fig. 4).
2. Microhardness test equipment (Fig. 5).
3. Sieve mesh 20 as a tool to coarsely separate fine charcoal after grinding giling (Fig. 6).

Figure 4. Furnance

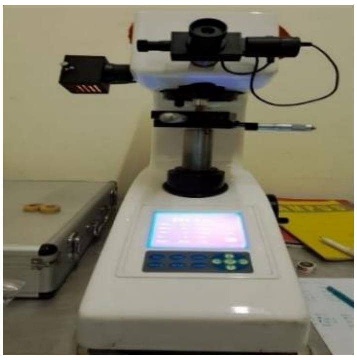

Figure 5. Microhardness test equipment

Figure 6. Sieve mesh 20

Figure 7. Sandpaper

Figure 8. Autosol and resin

Figure 9. Digital scales

Figure 10. Tensile load furnace

4. Sandpaper 50-5000 grit (Fig. 7).
5. Polishing; autosol and resin (Fig. 8).
6. Digital scales (Fig. 9).
7. Tensile load furnace (Fig. 10).

c. Research procedure

The research procedure for the pack carburizing process with the tensile method on carbon steel:

1. Carburizing process

1) Prepare a heat tensile test kit or heating kitchen (furnace).
2) The prepared specimen is put into a steel sleeve, which contains buffalo bone charcoal that has been mixed with $BaCO_3$ as designed, then the steel sleeve is closed and given a clay blockage on the part where there is a steel box gap, so that it is completely vacuum, the steel sleeve is inserted and arranged so that it is in the furnace.
3) Connect the furnace current with the heater to start the carburizing process.
4) Set the carburizing temperature after reaching the specified temperature 600^{o}C, 650^{o}C, 700^{o}C, and 750^{o}C, then hold for 4 hours in the furnace while being given a tensile load $1/4\sigma_p$, $1/2\sigma_p$, $3/4\sigma_p$, and σ_p.
5) Turn off the current to end the carburizing process and cool down in the furnace.
6) Then clean the surface of the object.
7) Perform the above steps with different variations (heating temperature) until all samples/specimens have been processed.

2. Micro Vickers hardness test

1) Prepare the sample: make sure the surface of the sample to be tested is flat and free of contaminants. If necessary, carry out surface smoothing using methods such as sanding or polishing.
2) Sample placement: place the sample under the indenter of the Vickers hardness testing equipment. Make sure the sample is in a stable position and perpendicular to the test instrument.
3) Hardness test: start the hardness test by pressing the indenter against the sample surface with a preset load. Indents will form on the sample surface.
4) Indentation measurement: once the indent is formed, measure the diagonal of the indent using a microscope attached to the Vickers hardness tester. The diagonal of the indent will be square, and measurements are taken to obtain the length of the diagonal.
5) Hardness value: the hardness value will be read on the monitor screen of the test equipment. Vickers hardness values are usually expressed on the HV (Vickers Hardness) scale.
6) Perform for hardness procedures on each test sample.

4.2. Research methods

This study is with a carburizing pack heated at heating temperatures below normal carburizing temperatures of 600^{o}C, 650^{o}C, 700^{o}C, and 750^{o}C while tensile loads of $1/4\sigma_p$, $1/2\sigma_p$, $3/4\sigma_p$, and σ_p (proportional stress). After reaching the heating temperature, the material is held in the furnace for 4 hours and after that, it is cooled slowly. After the material is cold, mechanical testing is carried out with Vickers microhardness. The Vickers method as a material hardness test is carried out by pressing the material or test specimen with a diamond indenter with a pyramid shape with a rectangular base and a large angle from the facing surface 136^o. Pressing with an indenter will produce a trace or indentation on the surface of the test material. To determine the value of the hardness of the

Table 2. Data from research on the hardness value of carbon steel under different furnace conditions

Specimens	Hardness (HRB)
Raw Material	55.8
Furnace laboratory (800^0C)	105.5
Furnace Tensile (800^0C)	105.2

Table 3. Data from research on the hardness value of carbon steel under carburizing conditions without tensile load

Heating Temperature (^{0}C)	Hardness (HRB)
800	105.5
750	101.5
700	94.1

Table 4. Data of the hardness test results at temperature 600ºC, 650ºC, 700ºC, and 750ºC

The temperature of Carburizing (ºC)	Hardness Brinell with Tensile Load			
	σ_p	$3/4\sigma_p$	$2/4\sigma_p$	$1/4\sigma_p$
600	103.93	93.87	89.77	85.97
650	104.33	99.13	90.07	85.47
700	104.80	101.20	93.53	93.27
750	106.60	101.63	96.67	95.60

test material, the average diagonal of the trace must be measured first by using a microscope.

5. Research Results

This research uses an experimental method, namely conducting experimental methods according to the carburizing method. The test data obtained in this study include the results of hardness testing on raw materials, hardness testing at a temperature of 800ºC on a laboratory-scale furnace without tensile and hardness testing on tensile load furnaces with loads of $11/4\sigma_p$, $1/2\sigma_p$, $3/4\sigma_p$, and σ_p, with heating temperatures of 600ºC, 650ºC, 700ºC, and 750ºC.

Research data for hardness in raw materials, laboratory furnaces and tensile load furnaces can be seen in Table 2 and 3. From the data in Table 2 and 3, it is obtained that the hardness value in laboratory furnaces with tensile load furnaces at the same temperature conditions, namely 800ºC without tensile load, gives the same value picture. This proves that the furnace condition at the tensile load is the same as that of a laboratory-scale furnace.

From Table 4 and Fig. 11, it can be obtained that the tensile load affects the hardness value of carbon steel at heating temperatures of 600ºC, 650ºC, 700ºC, and 750ºC where the highest hardness value is obtained at the tensile load of proportional stress and the lowest at the tensile load of $1/4\sigma_p$ temperature of 600ºC.

The highest value at the tensile load is proportional stress, namely at a temperature of 600ºC the hardness value is 103.93 HRB, at a temperature of 650ºC the hardness value is 104.33 HRB, the temperature of 700ºC the hardness value is 104.80 HRB, and at the temperature of 750ºC, the hardness value is 106.60 HRB, while the pack carburizing process without tensile at the heating

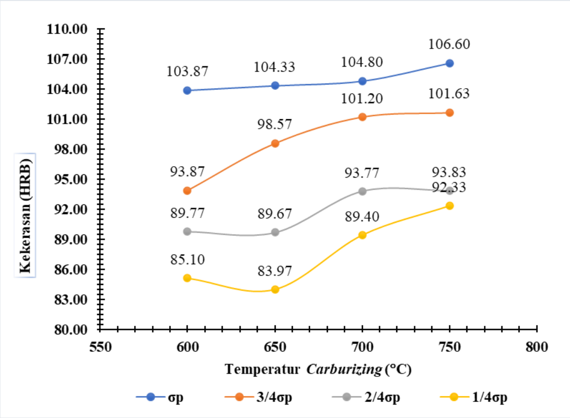

Figure 11. Graph of the effect of heating temperature on the carburizing process with a tensile load of $1/4\sigma_p$, $1/2\sigma_p$, $3/4\sigma_p$, and σ_p on the hardness of carbon steel

temperature of 800ºC the hardness value is 105.2 HRB. This proves that applying a load during the heating process at a lower heating temperature may exceed the hardness value without a tensile load at a higher temperature of 800ºC. From this, the carburizing heating temperature is minimized by applying a tensile load equal to the proportional stress. The process of applying loads in hot conditions causes strain which causes the distance between atoms to be greater so that carbon atoms in charcoal easily diffuse to fill the void and shift atoms in the specimen so that the carbon concentration increases.

The denser and increasing carbon atoms in the material result in the hardness will increase. This is in line with the results of research on that the diffusion of carbon causes the entry of carbon into the material and also based on the research of that with the tensile in the hot state, there will be a large strain rate and at a smaller temperature. With a large strain rate, the potential for interspace atoms is greater so that carbon atoms easily diffuse both by substitution and interstitial.

6. Conclusion

From the results of research on the effect of carburizing at temperatures of 600ºC, 650ºC, 700ºC, and 750ºC which are temperatures below normal carburizing temperatures, it was found that diffusion occurs that affects the hardness of carbon steel where the highest hardness occurs at 750ºC temperature with a tensile of proportional tensile of 106.60 HRB which passes the hardness value of the carburizing material at a temperature of 800ºC without a tensile load of 105. 2 HRB. This suggests that applying a drawing load equal to the proportional voltage of the material in the carburizing process can increase the hardness value even beyond the hardness value of carburizing without withdrawal due to the greater atomic interspace which allows the rate of carbon diffusion to occur easily.

Acknowledgements

Thank you very much to the Department of Mechanical Engineering, Hasanuddin University, South Sulawesi for contributing and supporting the publication of this research article.

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