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Effect of pack carburizing and viscosity of quenching media on AISI 1010 steel

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Abstract

Pack Carburizing is a method of surface hardening of steel which is diffusing carbon into the surface material where the carbon source is carbon active from charcoal. The objective of this research is to study the effect of the cooling media quenching type of pack carburizing process on microstructure characteristic of AISI 1010 steel. Moreover, it is also determining the effect of pack carburizing heat treatment such as Vickers hardness number to the mechanical properties of AISI 1010 steel. Characterization composition element use optical emission spectroscopy, metallography microscope to observe the microstructure and penetration of granular activated carbon as carburizing material on AISI 1010 steel surface. The temperature of pack carburizing is 950ºC in 3 hour and then specimen has tempering process after carburized in 870ºC with holding time for 60 minutes. This study uses 4 variations of engine oils cooling media in quenching process. Specimen R is raw material, specimen C is carburized specimen, specimen Q1 is specimen that quenched with engine oil SAE 0W-20, specimen Q2 is specimen that quenched with engine oil SAE 10W-40, specimen Q3 is specimen that quenched with engine oil SAE10W -60, specimen Q4 is specimen that quenched with distilled water. The results of the Vickers hardness test show that the average Vickers hardness number in specimen A is 365.29 ± 9.39 HVN, specimen B is 377.59 ± 24.12 HVN, specimen C is 397.94 ± 47.41HVN, specimen D is 475,90 ± 106.07HVN. The results of the composition test of specimen after pack carburized obtain the carbon content has significantly increase from 0.0987 wt%C to 4.1159 wt%C. The results of microstructure observations obtain that a massive pearlite structure was formed in average depth ± 0.794 mm on the surface of the pack carburizes specimen.

***Keywords**: carburizing, hardness, microstructure, quenching*

1. INTRODUCTION

Low carbon steel (<0.2 wt%) have been widely used as weight critical engineering structural materials, such as pressure vessel, steam generator and steel containment vessel, because of their toughness, sufficient strength, good weldability and low-cost environmentally efficiency (1). This research uses AISI 1010 low carbon steel, which is a type of steel that is familiarly used in industry as a construction material and various components of industrial equipment. In its application, low carbon steel must be increased its hardness, one of them is by carburizing. Low carbon steels tend not to be hardened by direct heat treatment such as by quenching because the carbon content is too low for significant transformation to occur. In order to successfully harden this grade of steel, carbon must be added to the surface of the steel, or commonly known as the carburizing process (2). The Method of carburizing is a process of hardening the surface of a metal object that having hard surface with compressive residual stress, it increases the hardness and fatigue life of a component (3,4). Carburizing is the process of saturating the surface layer of steel with carbon. Nowadays, in accordance with carbon source, carburization is classified as pack carburizing, gas carburizing and liquid carburizing. The main purpose of the carburizing process is to wear resistance surface on mechanical components by enrichment of the surface layer with carbon to a concentration from 0.75 to 1.2 % (5). Low carbon steels, containing from 0.1 to 0.18 % carbon, may be subjected to carburizing. The ability of carbon atoms to enter and insert into iron atoms in the form of a solid solution, causes the carbon content to increase and is used in the pack carburizing method. Then continued hardening by single-quenching fast cooling so that low carbon steels such as AISI 1010 can be improved for better hardness and can be used as an alternative material to replace medium carbon steel or high carbon steel.

Using heat with austenitic temperature between 850ºC to 950ºC, the carbon medium will be oxidized to produce CO_2 and CO gas. CO gas will react with the steel surface to form carbon atoms and then diffuse into the steel. The carbonization reaction can be explained as follows:

$$2C(s) + O_{2(g)} \rightarrow 2CO_{(g)} \quad (1)$$
$$BaCO_{3(s)} \rightarrow BaO(s) + CO_{2(g)} \quad (2)$$
$$CO_2(g) + C_{(g)} \rightarrow 2CO_{(g)} \quad (3)$$
$$2CO \rightarrow CO_2 + C_{(dissolved)} \quad (4)$$

Quenching is one of the stages of the heat treatment process, where quenching is the rapid cooling of metal to obtain the desired mechanical properties. Quenching is one of the hardening heat treatment processes, which has the aim of increasing the strength and hardness of steel by heating the metal at a certain temperature (845ºC - 870ºC), then cools the steel with a fast-cooling rate, resulting in a phase decomposition of austenite to martensite. In steel quenching process, the viscosity of the cooling medium has affected the microstructure formed. This microstructure change gives a different hardness value of a material. The cooling medium used in this research is Oil/Lubricant with Society of Automotive (SAE) standards. The phase formed in steel is affected by the cooling rate. This will certainly affect the mechanical properties of the steel. Many quenching media have been researched and applied in industry, including water, salt water, and oil. Viscosity is the level of viscosity possessed by a fluid. The higher viscosity index, the higher the rate of heat propagation that occurs. The viscosity of the oil as cooling media has affect influential in the cooling process of the specimen. Oil that has a lower viscosity has a better heat absorption ability than oil with a higher viscosity because the absorption of heat will be slower.

This study aimed to analyze the effect of pack carburizing on changes in the microstructure of AISI 1010 steel and then analyze the changes that occur in the microstructure and hardness of AISI 1010 when a single quenching process is carried out by varying the viscosity of the cooling media. Can the low carbon steel pack carburizing process be hardened and whether the pack carburizing results will have an effect on the microstructure and hardness values when quenched. The increase in hardness is expected to expand the application of AISI 1010 steel in industry.

2. Methods

The study used AISI 1010 low carbon steel as the test specimen. Pack carburizing was carried out at a temperature of 950ºC for 3 hours. Carburized mixture used is 80% Coconut Shell Charcoal and 20% Barium Carbonate ($BaCO_3$). Cooling is slowly decrease in the furnace. The single-quenching process by reheating the material to temperature in 870ºC and holding time for 60 minutes. The specimens were rapidly cooled with a variety of cooling media distilled water, several type of engine oil such as engine oil SAE 0W-20, engine oil SAE 10W-40, engine oil SAE 10W-60 and distilled water with kinematic viscosity index at 100ºC (ASTM D7279) is 8,163 cSt, 14,51 cSt, 22,94 cSt; and 0,28 cSt

Figure 1. Tool of Carburizing Process: (a) Barium Carbonate ($BaCO_3$);(b) Coconut Shell Charcoal; (c)Cooling medium; (d) Pack carburizing; (e) Vernier Calliper;(f) Microvickers hardness tester

2.1 Optical Emission Spectrometer-ASTM E415

The composition test was carried out using the Optical Emission Spectrometer (OES) - (ASTM E415) method on raw materials and pack carburizing results.

2.2 Standard ASTM E407

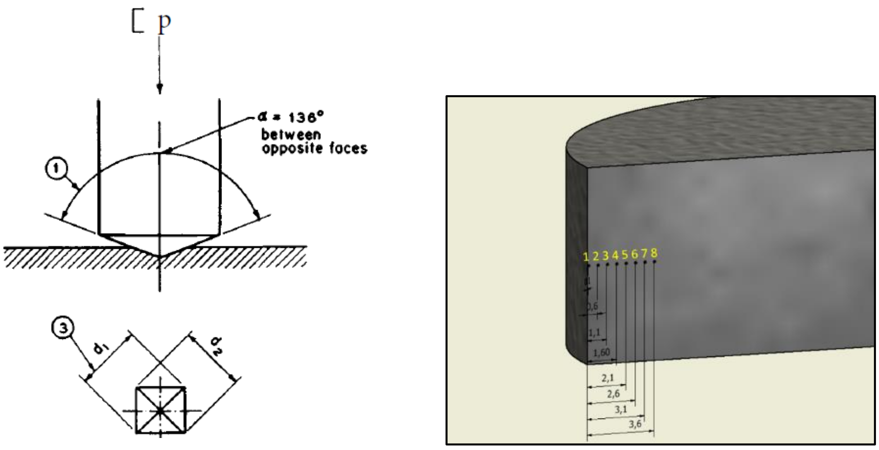
Changes in the microstructure were observed using an optical microscope with 500X magnification with nital HNO_3 2.5% (ASTM E407).

2.1 Standard ASTM E92-82

The hardness value of the material was tested by the Vickers method on the surface and micro-Vickers in the cross-section area. This test standard covers the determination of the Vickers hardness of metallic materials, using an applied load from 1 kgf to 120 kgf, the Vickers hardness number/Vicker's Hardness (HV) is a number related to the applied load and the surface area of the resulting trace of a square pyramidal diamond indenter having a face angle of 136^o, calculated from the equation:

$$HV=1.8544 \times F/D^2 \quad (5)$$

Where *HV* is notation Vickers Hardness Unit, *F* is notation Load (Kgf) and *D* is notation Average length of the diagonal indent (mm).



(a) (b)

Figure 2. Vickers Hardness Testing and Specimen: (a) Microvickers hardness testing methods ASTM E-92; (b) Indentation Points

3. Result and Discussion

3.1 Pack Carburizing

Composition material specimen has tested by Optical Emission Spectrometer. The results from composition test in Table 1. Si content has increased after pack carburized treatment from 0.2189% to 0.2199%, Mn content has increase from 0.3793% to 0.3915%, Cu content has increase from 0.0389% to 0.0413% and Ca content has increase from 0.002% to 0.0044%. Contents of Cr, P,Cu, Ni element has decrease after pack carburized

Table 1. Material Composition Test Results

Elements	Raw Material (%)	Carburized (%)
C	0.0987	4.1159
Si	0.2189	0.2199
Mn	0.3793	0.3915
P	0.0085	0.0047
Cr	0.0438	0.0316
Cu	0.0389	0.0413
Ni	0.0531	0.0519
Ca	0.002	0.0044
Fe	99.11	95.12

In Table 1, the carbon content has significant increase from 0.094% to 4.1159%. From these results it can be estimated that there is a deposit of carbon on the surface, which is possible to read due to the surface being less free from the pack carburizing scale or residual carbon that has slipped into the gaps in the steel surface formed due to the vacancy of elements that have a low evaporation point such as P, Cu, Ni, etc.). Again, that with the increase in carbon content, the initial indication of pack carburizing is successful.

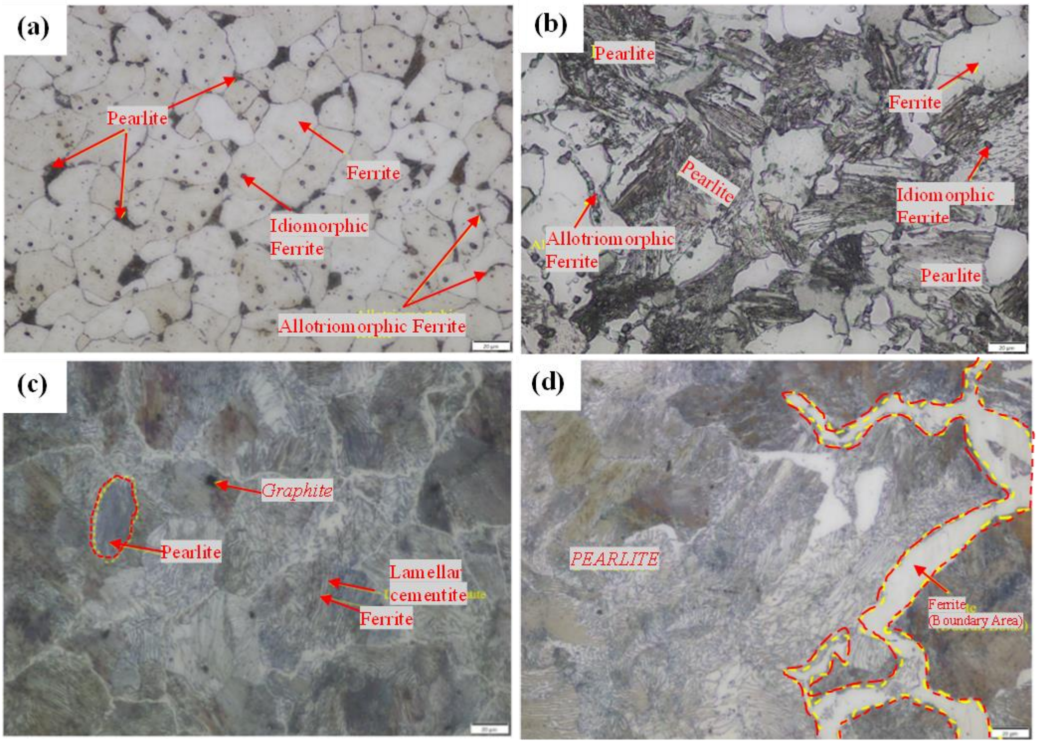


Figure 3. Changes In the Microstructure of Carburizing Results:
(a) Raw Material; (b) Carburizing Result; (c) Cross Section Specimen C Edge Area.
(d) Cross Section Specimen C Depth 0.6 mm; Magnification 500X ; Nital 2.5%

Observation of the microstructure of specimens R and C, which can be seen in Figure 2, where in Figure 2(a) is specimen R, it is seen that the dominant phase formed is Ferrite which is the phase of the element Fe. The Ferrite region is indicated by a white area due to the Fe element reacting with etching. It is shown that ferrite is also formed in Allotrimorphic and Idiomorphic forms. Allotriomorphic ferrite is formed at the grain boundaries of the ferrite region which is indicated by the thickening of the grain boundaries. While Idiomorphic formed in the Ferrite phase is indicated by small spheres that fill the grains. It was also seen in Specimen R that pearlite was formed in a minor manner.

In the microstructure of Specimen C, dark areas are seen, which indicates that the cementite (Fe_3C) structure was massively formed on the surface. Cementite is associated with the Ferrite phase to form a lamellar structure called the Pearlite phase. The pearlite structure is formed from the slow cooling of the specimen from the pack carburizing process. Massive formation of pearlite phase has showed in Figure 2(b) this phase has a higher hardness than the ferrite phase due to its higher carbon content and lamellar structure. Lath martensite structure is a typical quenched steel, coarse lath martensites surrounded by the ridge-like narrow laths have a generally wedged-shape and a high density of auto-tempered carbides, which is in accordance with the high Martensite start temperature of 673 K (6), Then the changes in the microstructure were reviewed by conducting a hardness test using the Vickers method on the surface of the material Table 2.

Table 2. Vickers Hardness Test Results

Specimens	Point	Hardness (HV)	Average (HV)	Standard Deviation
	1	177.642		
R	2	177.937	176.004	3.096
	3	172.432		
	1	220.789		
C	2	220.894	217.878	5.134
	3	211.950		
	1	376.073		
Q1	2	358.971	365.285	9.387
	3	360.811		
	1	379.152		
Q2	2	400.890	377.589	24.120
	3	352.725		
	1	343.240		
Q3	2	427.085	397.941	47.406
	3	423.499		
	1	365.703		
Q4	2	577.294	475.895	106.069
	3	484.689		

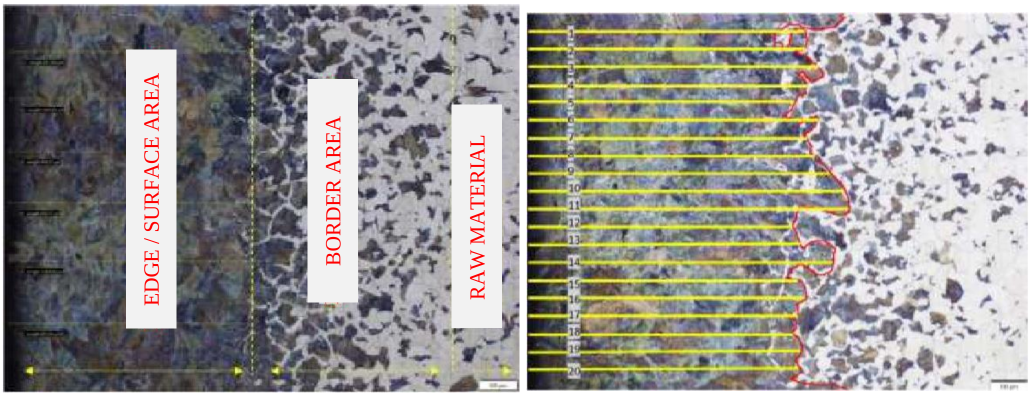


Figure 4. Microstructure of Cross Section Specimen C
(A: Carbon Penetration Area; B: Maximum Penetration Area); 200X ; Nital 2.5%

In Figure 4, it is found that the average border area occurs to a depth of ±0.794 mm. From Figure 4, shows that the carbon was successfully diffused to a depth of 1.1 mm from the surface of the material. The pearlite structure is massively formed to a depth of 0.7 mm. then to a depth of 1.1 mm is the boundary area, where the formation of pearlite begins to decrease, and the ferrite phase begins to appear again. In this border area, it shows the carbon diffusion limit resulting from pack carburizing. The lath martensite is a typical microstructure of low carbon and low alloy steels, which is formed by a diffusionless shear mechanism when austenite is cooled rapidly to room temperature.

Table 3. Carbon Penetration Depth Measurement Results

Label	Length (μm)	Label	Length (μm)
1	775	11	905.882
2	777.941	12	742.647
3	817647	13	758.824
4	783824	14	861.765
5	751471	15	764.706
6	804.413	16	780.882
7	779.412	17	748.529
8	805.884	18	744.118
9	848.529	19	769.118
10	885.294	20	764.706
Average		793.5296 μm 0.794 mm	

In Table 2, the characterization of the microstructure is in accordance with the results of the hardness tests carried out. Specimen R, which initially had an average initial hardness value of 176 HV, increased by 41.87 HV or 23.97% to 217.88 HV in specimen C. The review was then continued by looking at the cross-section area of the specimen surface (carbon diffused from the specimen). Microstructure observations and microhardness testing were carried out in the cross-section area. The observation of the microstructure in Figure 3 shows that the area on the edge/surface of specimen C is darker in color and forms pearlite grains, and then fades to the interior. This further supports that the carburizing pack succeeded in diffusing carbon from the environment into the specimen. Then the results of the micro cross section photo of specimen C were interpreted using ImageJ software. The carbon border area was then measured its distance from the edge of the material. This result can represent the average depth of maximum carbon diffused into the material.

Table 4. Microvickers Hardness Test Result

Point	Edge Distance (mm)	Hardness (HV)
1.	0.1	270.913
2.	0.6	263.736
3.	1.1	160.972
4.	1.6	119.328
5.	2.1	120.405
6.	2.6	118.265
7.	3.1	115.160
8.	3.6	113.157

Tested 8 points of hardness. Point 1 is the closest point to the surface of the material and point 8 is the inside/middle of the material. At 0.1 mm from the surface, the material has a hardness of 270.91 HV and in the area of 0.6 mm it has a hardness value of 263.74 HV. Then the hardness value began to decrease in the measurement results at a distance of 1.1mm from the surface where the hardness value was found to be 160.97 HV. At a distance of 1.6 to 3.6 mm, a relatively constant hardness value in the range of 117 HV indicates that the carbon does not diffuse to that depth. Bontong et. al. has study combine buffalo bone charcoal powder with $BaCO_3$ as carburizer, the hardness can reach 91.667 kg/mm^2 at a percentage of 80% BBP + 20% BaCO3. Can be concluded that the heat treatment of materials of low carbon steel at a diameter of 1, 588 cm with the pack carburizing at heating temperature 850°C with long lasting 30 minutes on the size of the mesh 30 and the load used for the suppression of 60 kg with the variation of the percentage of bone charcoal buffalo effect on the hardness (7),This further supports the analysis from Fig. 3 discussed earlier, that there is a hardness gradient in the material, or it can be concluded that the pack carburizing process was successful.

3.2 Single-Quenching

Microstructure observations and Vickers hardness testing were carried out on the quenched material with variations in the viscosity of the cooling medium. The cooling medium used is SAE 10W-60 Oil; SAE 10W-40 oil; SAE 0W-20 oil; and distilled water (distilled water), with kinematic viscosity values in units of cSt at a temperature of 100°C, respectively 22.94 cSt; 14.51 cSt; 8.163 cSt; and 0.28 cSt. Theoretically, the low viscosity index will affect the cooling rate in quenching.

Table 5. Submersion Temperature

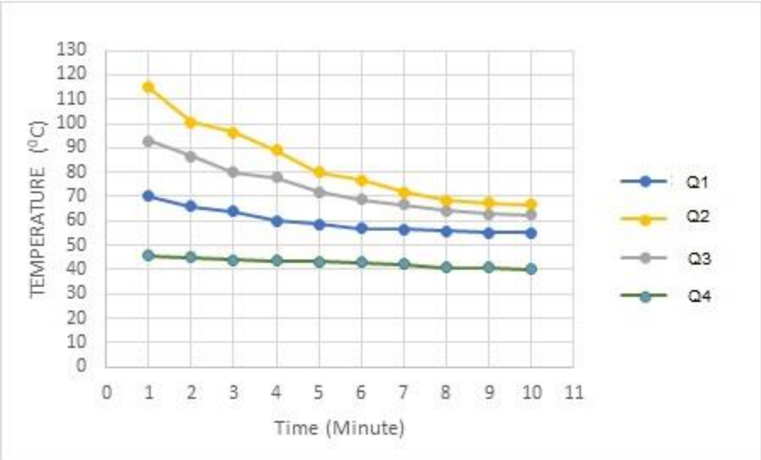
Time (minutes)	Temperature (°C)			
	Distilled water	SAE 0W20	SAE 10W40	SAE 10W60
0	870	870	870	870
1	70.3	92.7	114.8	45.8
2	65.9	86.4	100.4	44.9
3	63.8	80.1	96.4	44.1
4	60.2	77.8	88.9	43.6
5	58.5	71.8	80.1	43.3
6	57	68.6	76.7	42.8
7	56.4	66.7	71.9	42.2
8	56	64.4	68.3	41
9.	55.3	63	67.3	40.7
10	55.1	62.5	66.5	40.1
15	54.1	58.8	61.1	39.5
20	53,5	56,8	58,4	39
25	51,7	54	54,8	38,3
30	51,3	52,2	53	37,8
35	49,3	50,1	50,7	37,3
40	48,4	48,7	49	37,1
45	47,8	47,6	48	36,5
50	46,3	46,5	46,7	36,5
55	45	44,9	45,2	36,5
60	44,2	44	43,6	36,5

From the results of temperature measurements in Table 5 and Figure 3, it can also be seen that the cooling rate of the cooling media, where each cooling medium in every minute and every second experiences a different temperature drop.

Table 6. Cooling Rate

Media	10 Min		5 Min		3 Min		1 Min	
	°C /min	°C /sec	°C /min	°C /sec	°C /min	°C /sec	°C /min	°C /sec
SAE 60	80.35	1.34	157.98	2.63	257.87	4.30	755.20	12.59
SAE 40	80.75	1.35	159.64	2.66	263.30	4.39	777.30	12.96
SAE 20	81.49	1.36	162.30	2.71	268.73	4.48	799.70	13.33
Water	82.99	1.38	165.34	2.76	275.30	4.59	824.20	13.74

The rate of the temperature decrease depicted in Table 6. The data shows that in every 10, 5, 3, and 1 minute, the decrease is different. It means that the cooling medium temperature decreases during the submersion time. In addition, it can be concluded that the cooling medium with a lower viscosity has a faster cooling rate than the cooling medium with a high viscosity. When interpreted in graphical form, in the cooling medium with lower viscosity the temperature drop occurs to a lower temperature. Even for specimens Q4 at the 1st to 60-minute measurements have shown a sloping intention which means that quenching occurs very quickly in Fig. 3. Viscosity is very important to form the structure and also mechanical properties. The surface hardness and tensile strength increased, but the ductility of the specimen decreases after pack carburizing treatment, in proportion to the increase in temperature pack carburizing and content of BaCO3 in the carburizing agent (8).



Graph 1. Submersion Temperature during quenching

Microstructural observations were carried out with 500X magnification, resulting in different interpretations of each specimen. From this interpretation, broadly speaking, the changes that occur can be interpreted that each process can change the microstructure of the material. It can be observed in the Q4 specimen in Figure 4. The results of the microstructure observation on the Q4 material can be observed that there is a massive change in the microstructure of the material. The Q4 specimen, which was quenched with the lowest viscosity cooling medium, experienced the fastest cooling of the austenite temperature. From the observation of the microstructure of the Q4 specimen, it can be observed that the phase formed in the Q4 specimen is dominated by martensite. The carbon in the material is fully diffused in the authenticity phase when heated and then quenched which lowers the temperature rapidly to produce a new phase, namely martensite. The lath martensite is a typical microstructure of low carbon and low alloy steels, which is formed by a diffusionless shear mechanism when austenite is cooled rapidly to room temperature (9).

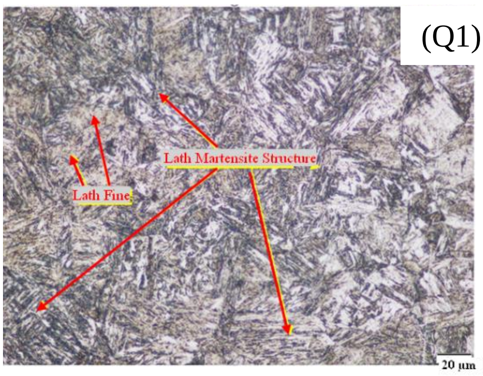


Figure 5. Microstructure of Q4 Specimen; 500X ; Nital 2.5%

This phase is a phase that is formed from the non-diffusion of carbon in the austenite crystal structure (FCC) when the temperature drops and forms a ferrite or pearlite (BCC) structure. This causes the carbon in the crystal structure to be trapped, causing the crystal structure to stretch. Strain due to the presence of the martensite phase causes inhibition at the grain location which means this martensitic structure will produce a harder material. In Specimen Q3, Q2, and Q1 the cooling speed decreases. This has an impact on the inability of the carbon in the austenite to completely transform into martensite. In the dark colored region, it is very possible that the phase formed is the martensite phase. This is supported by the less lamellar structure found when compared to specimen C. The study of carburizing has effects to the microstructure such Jiang et. al. the martensite structure has start cooling rate and temperature at the surface are 0.05 °C/s and 200 °C, respectively. The martensite start temperature of the steel at the matrix is nearly 320 °C. The ‘‘nose’’ of the TTT curve of austenite to bainite transformation of the steel at the matrix is 400 °C (10).

Figure 5. The cooling speed is still quite high resulting in the dominance of the martensite phase and the slower the cooling rate, the more the pearlite phase is formed in lamellar form. Associated with hardness, the hardness of the martensite phase has the highest hardness, then pearlite, and ferrite which is the phase of the element Fe. However, due to heating to the austenitizing temperature and then rapidly cooling has affect the Widmanstatten structure is formed. In the sudy of microstructure refinement has affect to mechanical properties such as research that Zhu.et. al. the microstructure reinement by the austenite films is very effective in improving the resistance to cleavage fracture by introducing barriers to crack propagation (11).

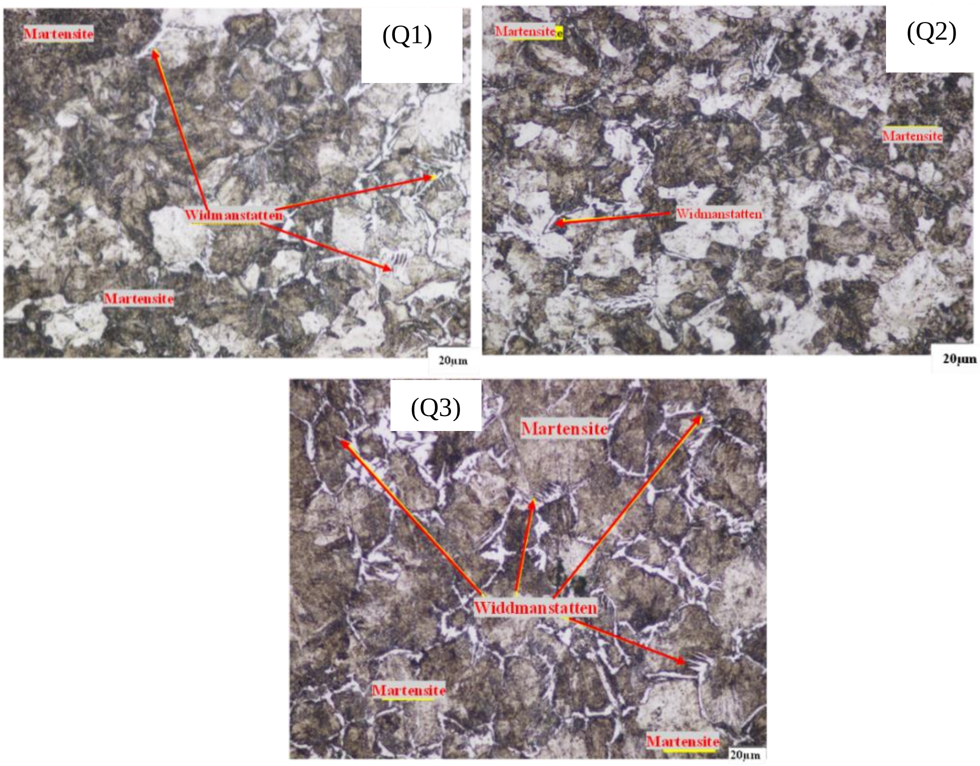
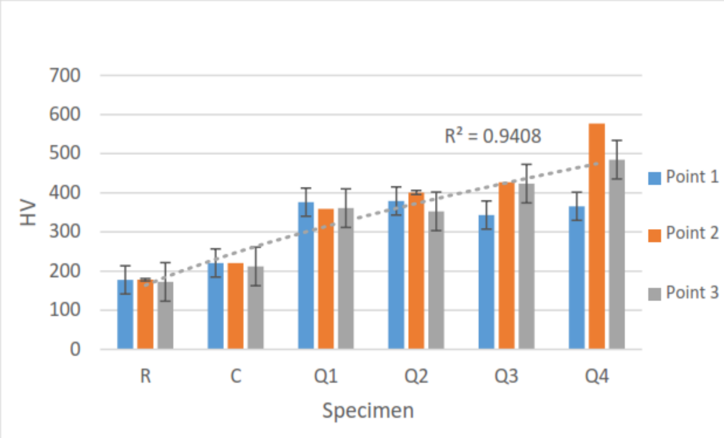


Figure 6. Microstructure of Specimen Q1-Q2-Q3 ; 500X ; Nital 2.5%

In Figure 6 we can compare the differences in the microstructures of Q1, Q2, and Q3 which have relatively similar structures. Microstructure observations were carried out to predict the mechanical properties of the metal. In general, as stated in the previous section, the single quenching process with variations in viscosity in this study succeeded in producing an increase in hardness based on the microstructure formed.



Graph 2. Vickers Hardness Test Results

In the According from Amos et.al. carbon martensite is different from that of pearlite in high carbon martensit and spheroidization of pearlite can be divided into two stages: The fragmentation of cementite laminates and the coarsening of cementite particles (12), The different results as formation of cementite laminates has different form because the carbon content and temperature parameter compare with low carbon AISI 1010.

The effect of this difference in viscosity is then supported by the results of the hardness test which can be seen in Table 2. Shows the difference in the value of the hardness differences in Q3-Q2-Q1 is not too significant. The average hardness value of Q3 is 397.94±47.41 HV, higher than Q2 which has a hardness value of 377.59±24.12 HV, and Q2 which is higher than Q1 which has a hardness value of 365.29±9.39 HV. These data still support that the hardness, in general, continues to change which is in line with the decrease in the viscosity of the cooling medium. Several test points were found that were not in accordance with the theory, for example, point 1 in Q4 and Q3 was even lower than the average hardness value of Q1. Significant hardness has increase on the surface of steel heat- treatment and shot speening. Experimental and analytically calculated S-N curves at 4-point cyclic bending quantify the individual effects resulting from the applied heat treatment and shot peening on fatigue life (13). The crack always grew directly into the matrix, he microstructure in the matrix of the carburizing steel was low carbon martensite with a superior toughness to the medium carbon martensite at the transition zone (14). Rui, et. al. has studied the effect of hardening mechanism of quenching tempering in carburized steel has obtain that at the first stage heat quenching tempering preservation was conducted at 740 °C for 3 h, in the second stage, it was insulated at 700 °C for 2.25 h. Hardness of sample spheroidizing-critical annealing is 302 HV (15). This can occur due to the tracking point when the Vickers hardness test is in the phase region with a low hardness value. This also occurs at point 3 of the Q4 specimen which has a very high hardness value compared to other points, which is possible at this tracking point the area affected by the indenter is dominated by a hard phase such as martensite. The increase in hardness is presented in the form of a graph which can be seen in Figure 6.

4. Conclusion

As Packed carburizing was carried out at a temperature of 950^{o}C for 3 hours. Carburized mixture used is 80% w/w Coconut Shell Charcoal and 20%w/w Barium Carbonate ($BaCO_3$) and quench temperature is 870^{o}C and holding time for 60 minutes. The microstructure and composition element has been studied by optical emission spectroscopy, metallography microscope to observe in AISI 1010 steel and also mechanical properties such as hardness vickers number. The main conclusions are summarized as:

1. Pack carburizing was successfully carried out on AISI 1010 steel, as indicated by the increase in carbon and several elements content such as Si content has increased after pack carburized treatment from 0.2189% to 0.2199%, Mn content has increase from 0.3793% to 0.3915%, Cu content has increase from 0.0389% to 0.0413% and also Ca content has increase from 0.002% to 0.0044%in the test material and the

effect on changes in microstructure with pearlite formation on the surface area to a depth of ± 0.794 mm. This change in microstructure such as lath martensite also is a typical microstructure of low carbon and low alloy steels.

2. The single quenching process provides changes to the microstructure formed on carburizing AISI 1010 steel, with the formation of a Martensite phase on the steel surface which then results in an increase in the hardness value. Variations in the viscosity of the quenching media produced the highest hardness obtained by cooling media with the lowest kinematic viscosity values in units of cSt at a temperature of 100ºC, namely distilled water/distilled water has kinematic viscosity index at 0.28 cSt, then SAE 0W-20 is 8.163 cSt, SAE 10W-40 is 14.51 cSt, and SAE 10W-60 oil is 22.94 cSt, with an increasing hardness to 475.90±106.07 HV, respectively; 397.94±47.41 HV; 377.59±24.12 HV; and 365.29±9.39 HV or an increase of 170,39%; 126.10%; 114.53%; and 107.54% hardness of Specimen R or raw material, there is decrease value after pack carburizing with distilled water as media quenching.

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