UDC 669
DOI: 10.15587/1729-4061.2021.228627

A STUDY OF THE PACK CARBURIZING QUENCHING TREATMENT WITH CANE MOLASSES COOLING MEDIUM EFFECT ON THE WEAR RESISTANCE OF LOW CARBON STEEL

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In the present study, various quenching media were added as cooling media for the quenching after pack carburizing treatment. The aim of this research is to get a suitable cooling medium for pack carburizing quenching treatment to increase the wear resistance of low carbon steel. Many cylindrical specimens for the adhesion wear tests were prepared from the used SS400 steel according to ASTM G99-04 specifications. Two heat treatment processes, namely pack carburizing and quenching were done. Firstly, the specimens are pack-carburized at a temperature of 875 °C, soaking time of 2 hours and quenched. The carburizing agent consists of Pinctada maxima shell powder (PMSP) and corn cob charcoal with a weight ratio of 30:70 %. Different cooling media (water, 10 % NaCl solution, 10 % cane molasses) in the pack carburizing quenching treatment are subjected to different kinds of tests. The hardness test was performed using Vickers micro hardness tester, the wear resistance was used in adhesive wear test, the carbon content was determined and microstructure examination was made using a scanning electron microscope (SEM-EDX). The result showed that all cooling media contributed to an increase in mechanical properties (surface hardness number, wear resistance), carbon content and microstructure change. The use of cooling media in the pack carburizing quenching process generally increases the surface hardness number of the specimen. The highest surface hardness number was 595 kg/mm2, respectively using 10 % cane molasses. The work shows that cane molasses can be used as a cooling medium for pack carburizing quenching of SS400 steel and contributed to the improvement of wear resistance

Keywords: low carbon steel, SS400 steel, hardness test, wear test, SEM-EDX, cooling medium, pack carburizing, quenching, surface hardness number, wear resistance

Received date 07.02.2021
Accepted date 11.04.2021
Published date 30.04.2021

How to Cite: *Darmo, S., Sinarep, S., Soenoko, R. (2021). A study of the pack carburizing quenching treatment with cane molasses cooling medium effect on the wear resistance of low carbon steel. Eastern-European Journal of Enterprise Technologies, 2 (12 (110)), 32–37. doi: https://doi.org/10.15587/1729-4061.2021.228627*

1. Introduction

The structural steel (SS400 steel) is a group of engineering low carbon steel materials used in automobile bodies, tin plates, panels, wire products, seamless tubes and agricultural tool products, etc. due to their specific properties such as high ductility, low surface hardness number, weldability and malleability. However, most of the engineering components are subject to wear. Significant economic losses occur due to wear in the mechanical engineering components. Surface hardness number affects wear resistance [1]. Wear resistance is classified into many types such as adhesive wear, which occurs when two surfaces move relative to one another, and these are relative movements in one direction or sequential movements under the influence of loads so that the stresses on the projections are large enough to create deformation and plastic adhesion. This adhesion will be at a high level of efficiency and capability in a clean environment and the area will increase during movement. Eventually, there will be some relative wear on the superficial network at the weak point of the visible place [2].

Low carbon steel has α phase with body-centered cubic structure, which transforms to γ phase with face-centered cubic structure at 910 °C. The low carbon steels contain less than 0.30 % C with manganese contents maximum of 0.40 [3]. The type of crystal structure is ferrite, which is soft, has a low surface hardness number, so that the wear resistance is short. Unfortunately, this also makes the steel ductile and less wear-resistant than medium or high carbon steel [4]. So its tribological properties should be developed when it is subjected to wear conditions. Those properties can be obtained by surface modification [5], with carbon diffusion increases its content on low carbon steel surfaces.

The urgency of the topic of this research is that the pack carburizing treatment of low carbon steel carried out by modern industry only adds carbon content to the steel surface and surface hardness has not considered changes in microstructure (still in the form of ferrite structure) and wear resistance. So it is very necessary to perform pack carburizing quenching treatment with a variety of cooling media, to produce changes in the ferrite microstructure to pearlite, so that surface hardness and wear resistance also increase.

The results of this study are expected to be used in the cutting tool manufacturing industry and the agricultural equipment industry. The resulting product has better quality in addition to a hard, sharp surface, as well as longer wear resistance. The use of cane molasses cooling medium is more environmentally friendly and does not cause health-causing odors compared to oil, which is commonly used in the quenching process in modern industry.

2. Literature review and problem statement

Carburizing surface treatment or carburization is a heat treatment process in which the surface composition of the low carbon steel changes by diffusion of carbon and results in increased surface hardness number with good wear resistance. It has been shown that using surface treatment engineering materials increases the wear resistance, decreases friction coefficient, and improves corrosion resistance [6, 7].

Pack carburizing is commonly used to increase wear resistance and impact toughness of low carbon steels by increasing the carbon content of the surface. The work [8] investigated the effect of carburizing time and carburizing medium on the microstructure, hardness and case depth of low carbon steel. The samples carburized using carbon black nanoparticles showed higher hardness than the samples carburized using acetylene gas or charcoal-based carburizing mixture.

Comparative study of wear resistance of low carbon steel after pack carburizing using different media was done [1]. The influence of various carburizing compounds, such as charcoal, cow bone, $CaCO_3$ added as energizer, on wear resistance was investigated. Three heat treatment processes were done, namely pack carburizing, quenching, and tempering. The result showed that all carburizing compounds contributed to increasing wear resistance and the compound of cow bone with 10 % $CaCO_3$ as an energizer had a carburizing case depth of 2.32 mm, which gives the highest wear resistance, while the charcoal compound gives a case depth of 1.1 mm.

Recently, different surface modification methods such as laser carburizing and nitriding, plasma carburizing, oxidizing and oxinitriding, gas carburizing and nitriding and glow discharge methods were used to improve the tribological, fatigue parameters and surface hardness number of this steel [9]. The work [5] investigated the stability of expanded austenite, generated by ion carburizing and ion nitriding of AISI 316L SS under high temperature and high energy pulsed ion beam irradiation. In both cases, the resulting FCC crystal structure, supersaturated with nitrogen or carbon, is strongly hardened with improved wear resistance, while maintaining the original corrosion resistance. The enhancement of fatigue wear resistance of gray cast iron by localized laser carburizing is discussed in detail in [9]. They showed laser carburizing is a more effective means of improving wear resistance than laser remelting and that the improvement is significantly affected by an increase in energy density during treatment. The effect of plasma carburizing and DLC (diamond-like carbon) coating on friction wear characteristics, mechanical properties and fatigue strength of stainless steel is investigated in [7]. They also showed that the DLC layer was markedly effective in decreasing the friction coefficient and improving wear resistance and furthermore, fatigue strength was greatly improved by hybrid surface treatment.

In spite of many advantages of ion carburizing, gas carburizing, plasma carburizing and laser carburizing processes, they are expensive, have complicated equipment and high treatment times and in some situations, they can cause the formation of deleterious phases and residual stresses [10]. Pack carburizing is a less expensive and much simpler process to modify the surface of low carbon steel [11].

The effect of the cooling rate of cane molasses as a quenching medium for 0.61 % C high carbon steels is considered [12]. Samples of high carbon steel were spheroidize-annealed and then machined prior to the hardening process. Molasses solution of viscosity equal to that of engine oil was prepared by adding water. The research showed that cane molasses can harden high carbon steel without cracking the component in the same way as engine oil, hence, molasses could be a very good alternative to engine oil for use as a quenching medium.

Up to now, no report about pack carburizing quenching of SS400 low carbon steel was observed in the open literature. Therefore, in this research, the pack carburizing quenching process was performed on SS400 steel to modify the surface by improvement of hardness to enhance wear resistance.

3. The aim and objectives of the study

The aim of the study is to determine the effect of the pack carburizing quenching process with cane molasses medium on the wear resistance of SS400 low carbon steel. This will allow evaluating the effects of various quenching media, such as water, NaCl solution, cane molasses added as a cooling medium for the quenching after pack carburizing treatment.

To achieve this aim, the following objectives were set:

– to determine the carbon content;

– to observe microstructure changes;

– to find out the distribution of the surface hardness number;

– to determine the wear resistance of the specimens after the pack carburizing quenching process with variations of the cooling medium.

4. Material and methods of the study

In the present study, SS400 low carbon steel with the chemical composition of 98.34 % Fe, 0.17 % C, 1.4 % Mn, 0.045 % P and 0.045 % S (all in wt. %) was used as the samples, and it was carried out in laboratory room temperature conditions. This steel was produced by forging process at the forged temperature of 560 °C with a 60 % reduction in area. SS400 steel is cylindrical with a diameter of 10 mm and is cut to a length of 20 mm for the surface hardness test and adhesion wear tests according to the ASTM (G99-04) standard.

Before testing the specimens, they were given a pack carburizing surface treatment followed by quenching. The specimens of SS400 low carbon steel were placed in a carburizing box (with dimensions of 230×200×123 mm^3), cut from wrought plate and covered by a carburizing agent. It consists of Pinctada maxima shell powder (PMSP) and corn cob charcoal with a weight percentage ratio of 30 %:70 %. Charcoal used as a mixture of the carburizing agent is corn cob charcoal. Corn cobs originated from Hybrid Petro Hi-Corn, which was planted by farmers in West Nusa Tenggara, Indonesia. The corn cobs are made of charcoal by burning, then milled with a milling machine to obtain a uniform corn cob charcoal, with a uniform particle size of 0.15 mm.

Pack carburizing quenching was done in an electric furnace (Carbolite RHF 1700). Firstly, the specimens are carburized at 875 °C for 2 hours as soaking time. Heating rates from room temperature to 500 °C and from 500 °C to the final heating temperature were set as 10 °C min^{-1} and 5 °C min^{-1}, respectively. The next specimens were quenched with different cooling media (water, 10 % NaCl solution, 10 % cane molasses). After completion of the treatment, tests include composition tests, microstructure observations, surface hardness, and wear resistance testing, with categorization of specimens as shown in Table 1.

Table 1

Categorization of pack carburizing quenching test specimens

Specimen	Condition of specimen
A	As received
B	pack carburizing quenching (medium cooled water)
C	pack carburizing quenching (medium cooled 10 % NaCl solution)
D	pack carburizing quenching (medium cooled 10 % cane molasses)

Microstructural changes and carbon content were studied with SEM-EDX (Cam Scan MV2300-Canada) equipped with energy dispersive X-ray (EDX), respectively. The etched specimens were placed under a microscope on the SEM-EDX. So that the measurement of the thickness of the carbon layer can be done because it is inside the eyepiece lens the microscope has a measurement scale in micron meters (1/1,000 mm). The total magnification of the microscope used is 400× consisting of 10× magnifying lens magnification and 40× objective lens magnification.

Hardness tests were carried out using Vickers micro-hardness tester (HV-1000-Canada) using 1,000 g of penetration load.

Adhesive wear test was implemented for all specimens in Table 1 and using the pin-on-disc method as shown in Fig. 1. The specimens were fixed by the bearer in a vertical position on a steel disc having a hardness of 58 HRC and rotated at 940 rpm. Adhesive wear test is conducted to determine the wear rate and wear resistance of the specimens.

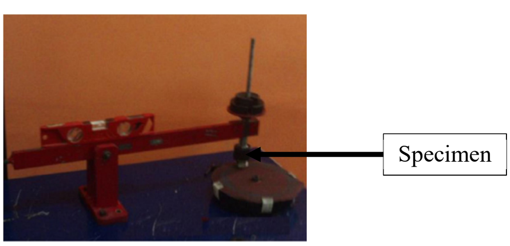

Fig. 1. Adhesive wear test equipment

Wear test is carried out to predict the wear performance and to investigate the wear mechanism. The test is performed to evaluate the wear property of the material so as to determine whether the material is adequate for a specific wear application. Adhesive wear is a type of wear due to localized bonding between contacting solid surfaces leading to material transfer between two surfaces or loss from either surface. Adhesive wear is the second most costly type of wear following abrasive wear. Abrasion is more common and apparent, but adhesive wear often turns into abrasion. Adhesive wear is caused by localized bonding between opposing surfaces.

5. Results of research

5. 1. Determining the carbon content

Based on the composition analysis with SEM-EDX, there was an increase in carbon content in the specimens after the pack carburizing quenching treatment. Significant changes occurred in the pack carburizing quenching treatment at a temperature of 875 °C, soaking time of 2 hours, with 10 % cane molasses cooling medium, shown in Table 2. The carbon content in the specimens (SS400 steel) in general changed. An increase in the percentage of carbon content in the specimens treated by pack carburizing quenching is 0.21 %, 0.35 % and 0.57 % for the use of water, 10 % NaCl solution and 10 % cane molasses cooling media, respectively.

Table 2

Carbon content of SS400 low carbon steel after the pack carburizing quenching treatment

Element Type	Quenching Medium		
	Water	10 % NaCl Solution	10 % Cane Molasses
C	0.21	0.35	0.57
O	18.38	19.04	18.82
Mn	0.32	0.32	0.32
Fe	80.29	80.29	80.29

Based on the data in Table 2, the carbon content in the specimens increases (the carbon content was originally 0.17 %). Changes in carbon content are influenced by variations in the use of cooling media in the carburizing pack quenching treatment. Changes are caused by differences in the viscosity of the three types of the cooling medium. The highest change in carbon content is in the use of 10 % cane molasses cooling medium. Because the viscosity is the highest, it causes the highest cooling speed compared to the cooling media water and 10 % NaCl solution. It has been known that the change of the carbon content is caused by the cooling rate in the quenching treatment, and the cooling rate is influenced by the viscosity of the cooling medium [10, 12].

5. 2. Observation of the microstructure

The specimens for metallographic analysis were prepared according to the ASTM E3 standard and etched in a 2 % nital solution. The microstructure of the specimens after treatment was studied with the help of SEM-EDX. Fig. 2 shows SEM-EDX observation of the surface of the sample treated by pack carburizing quenching at a temperature of 875 °C, soaking time of 2 hours with a variation of cooling medium. The microstructure change on the specimen surface after pack carburizing quenching treatment with variation of cooling medium: water medium, 10 % NaCl medium, and 10 % cane molasses is shown in

Fig. 2, *b–d*. In Fig. 2, *a*, the microstructure is shown, the initial (untreated) specimen is ferrite. Ferrite is a metal composition that has a maximum carbon solubility limit of 0.025 % carbon at a temperature of 723 °C, its crystal structure is BCC (Body-Centered Cubic) and at room temperature has a carbon solubility limit of 0.008 % carbon. After the pack carburizing quenching treatment with water cooling medium, pearlite begins to form. Likewise with the use of cooling medium C (10 % NaCl solution) and D (10 % cane molasses). The difference is in the shape of the granules. Pearlite formed in specimen B (water medium) has a finer grain structure compared to specimens C and D, as shown in Fig. 2, *b–d*. The difference in grain size in the pearlite structure is due to the use of different cooling media in the pack carburizing quenching process. The difference in viscosity between the water cooling media, 10 % NaCl solution and 10 % cane molasses causes a difference in cooling speed, so that the size of the granules formed is different. The greater the cooling speed, the finer the pearlite structure formed, the smaller the cooling speed, the coarser the pearlite structure formed.

Therefore, according to Fig. 2, there is a change in the structure of ferrite to pearlite due to heating that exceeds the critical temperature of AC1, which is around 723 °C [5]. Pack carburizing treatment is carried out at a temperature of 875 °C. Furthermore, the change in grain size in the pearlite structure is influenced by the cooling medium used in the quenching treatment.

5. 3. Distribution of the surface hardness number

We measured the surface hardness number using a method of Vickers hardness number. The basic principle of the Vickers hardness testing method is to press the specimen with a diamond indentor in the form of a pyramid with a rectangular base and a large face-facing angle of 136°. The indentor compressive load causes a trace on the surface of the test object in the form of a curve. The data from the test results are in the form of the width of the diameter of the pressure marks with an indentor, then the results are plotted into a graph, shown in Fig. 3.

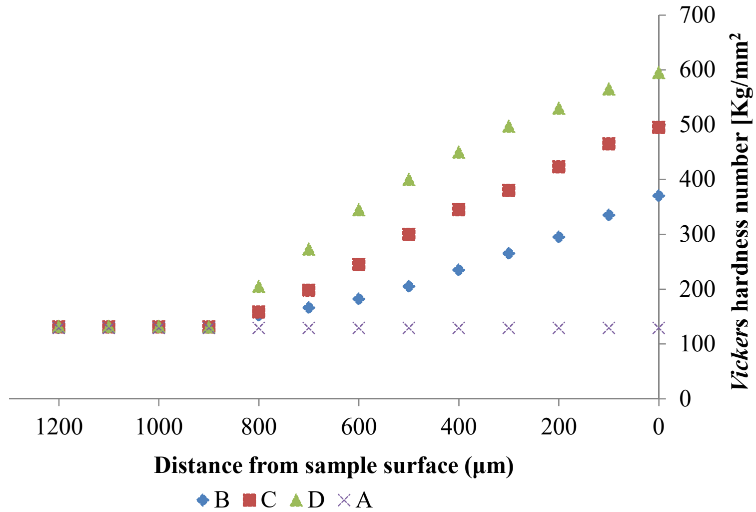

Fig. 3. Distribution of the surface hardness number of specimens for pack carburizing quenching at a temperature of 875 °C and soaking time of 2 hours

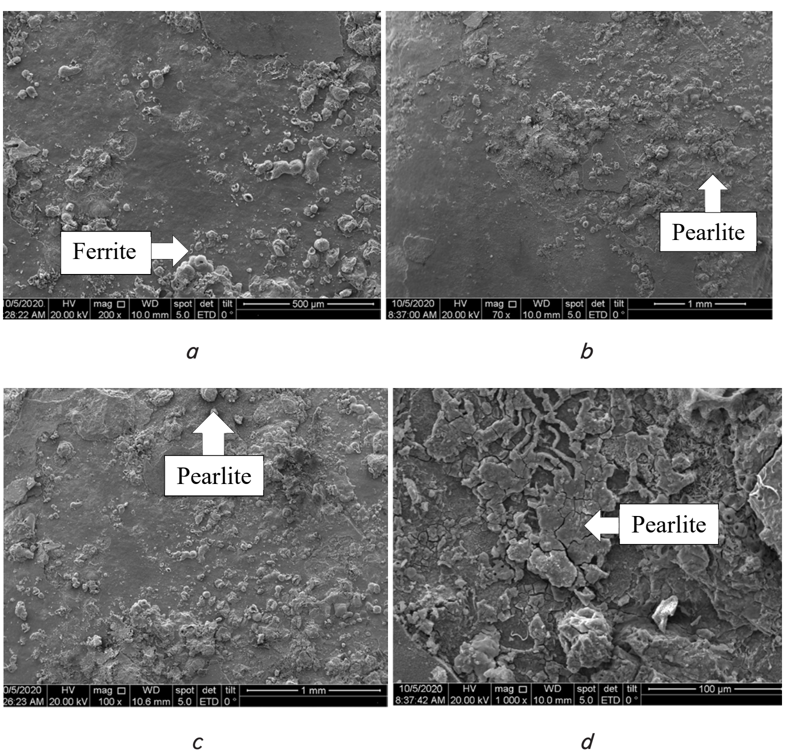

Fig. 2. Microstructure of the surface layer for pack carburizing quenching at a temperature of 875 °C, soaking time of 2 hours, with a variation of cooling medium: *a* – untreated specimen; *b* – water medium; *c* – 10 % NaCl solution; *d* – 10 % cane molasses

Fig. 3 is the result of the study of the surface hardness number. It shows the results of the distribution of the surface hardness number for pack carburizing quenching at a temperature of 875 °C, soaking time of 2 hours. After the treatment, the surface hardness number of the specimens increased. The increase in the surface hardness number occurred from 0 to 900 μm from the surface of the specimens. The farther away from initial (untreated) specimens, the surface hardness number didn't increase, from the surface until the core of specimens. The surface hardness number is 129 Kg/mm^2. The use of cooling media in the pack carburizing quenching process generally increases the surface hardness number of the specimen. The surface hardness numbers were 370, 495, and 595 Kg/mm^2, respectively for the use of cooling medium water, 10 % NaCl solution, and 10 % cane molasses. The difference in hardness number is caused by the difference in viscosity, which has an impact on the difference in cooling speed. The higher the viscosity, the slower the cooling speed, so that the growth of pearlite grain boundaries is larger (coarse) than for the less viscous cooling medium. The order of viscosity of the cooling medium used from lower to higher is water, 10 % NaCl solution and 10 % cane molasses cooling media.

5. 4. Determining the wear resistance

The wear resistance test is carried out with a constant compressive load of 2 Kg and a sliding speed of 5 m/sec. The results of the wear rate test of specimens subjected to pack carburizing quenching are shown in Fig. 4 and the wear resistance is shown in Fig. 5. The relationship between wear rate and its time parameters is shown. The wear rate increased when time increased and this was clear in all specimens of group A, B, C, D, and E but the wear resistance decreased since it depends on hardness. Fig. 4, which represents the relationship between time and wear rate shows that specimen A (untreated specimen) gives the highest wear rate equal to 11.14 mg/cm while specimen E (treated by pack carburizing quenching using 10 % cane molasses cooling medium) gives the lowest wear rate equal to 2.86 mg/cm for a wear resistance test time of 40 minutes.

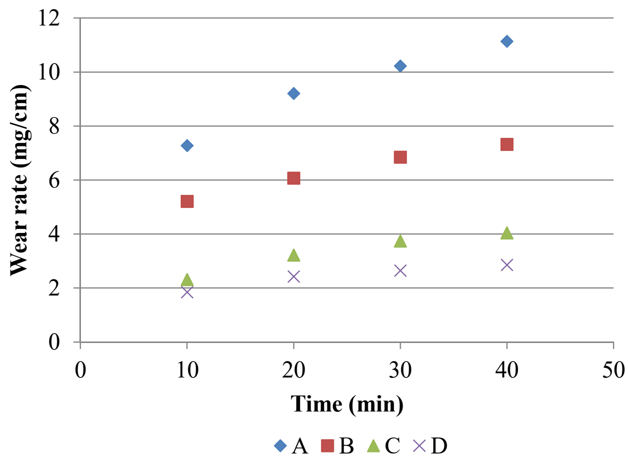

Fig. 4. Wear rate of specimens for pack carburizing quenching at a temperature of 875 °C and soaking time of 2 hours

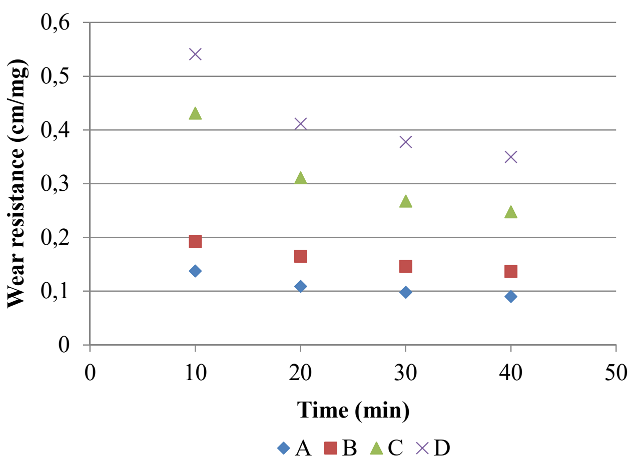

Fig. 5. Wear resistance of specimens for pack carburizing quenching at a temperature of 875 °C and soaking time of 2 hours

Generally, the cooling media (water, 10 % NaCl solution, 10 % cane molasses) used in the pack carburizing quenching treatment affect wear resistance. Wear resistance is increased compared to initial (untreated) specimens.

Fig. 5 shows the effect of cooling media on the wear resistance of specimens after pack carburizing quenching treatment. It represents the relationship between time and wear rate and shows that specimen E gives the highest wear resistance equal to 0.35 cm/mg while specimen A gives the lowest wear rate equal to 0.09 cm/mg, for a wear resistance test time of 40 minutes.

6. Discussion of pack carburizing quenching treatment using the cane molasses cooling medium effect on the properties of low carbon steel

The wear resistance of SS4400 low carbon steel is related to the surface hardness figure [1, 4]. Surface hardness rate is influenced by carbon content and microstructure after heat treatment and cooling processes [5]. Therefore, cane molasses cooling medium is used in pack carburizing quenching treatment.

The result of pack carburizing quenching treatment was the diffusion process, which caused changes in the carbon content, microstructure on the specimens surface, which also resulted in an increase in the surface hardness number and wear resistance of the specimens. The diffuse carbon element originated from carburizing agent consisting of corn cobs charcoal and Pinctada maxima shell powder (PMSP). Corn cobs charcoal is a source of carbon and PMSP is a source of calcium carbonate ($CaCO_3$) as an energizer that accelerates the diffusion process [11]. Pinctada maxima is a type of mollusk that has a protective shell, containing calcium carbonate, which is discussed in detail in [10, 13]. Diffusion occurs according to the following chemical reactions. At high temperatures (800–900 °C (carburizing temperature)), Fe_3C (carbon steel) and $CaCO_3$ will decompose through the reaction:

$$Fe_3C \Leftrightarrow 3Fe + C.$$

$$CaCO_3 \Leftrightarrow CaO + CO_2.$$

This carbon dioxide finally reacts with C from steel with the following reaction:

$$CO_2 + C \Leftrightarrow 2CO.$$

$$2CO + O_2 \Leftrightarrow 2CO_2.$$

CO gas will change to CO_2 and the reaction will continue. Based on the phenomenon obtained in this study, there is a process of carbon diffusion from the carburizing agent to the surface of the specimens. The process is commonly called pack carburizing, conducted in [1].

In Fig. 2, *a*, the microstructure is shown, the initial (untreated) specimen is ferrite. Ferrite is a metal composition that has a maximum carbon solubility limit of 0.025 % at a temperature of 723 °C, its crystal structure is BCC (Body-Centered Cubic) and at room temperature has a carbon solubility limit of 0.008 %. After pack carburizing quenching treatment with water cooling medium, it changes to a pearlite microstructure. Pearlite is a mixture of eutectoid between ferrite and cementite, which is formed at a temperature of 723 °C with a carbon content of 0.83 % Cementite is a compound consisting of the Fe and C elements with a certain ratio with the chemical formula Fe_3C and its crystal structure is orthorhombic [3].

The wear rate increased when time increased and this was clear in all specimens of group A, B, C, D and E but the wear resistance decreased. Fig. 4, which represents the relationship between time and wear rate shows that specimen A (untreated specimen) gives the highest wear rate while specimen E (treated by pack carburizing quenching using 10 % cane molasses cooling medium) gives the lowest wear rate. The phenomenon shows the effect of the second parameter (load) on wear rate. It caused an increase in the plastic deformation in surface tips peaks between two sliding surfaces. The adhesive process of the two tips surfaces depends on applied load. If the load is low, the contact appears in the upper bit and this was very thin during the sliding process, which causes a thin oxide layer to work as a protective surface film, which limits the touching between the two sliding surfaces and prevents the direct metallic connection between the surfaces tips. Thus, the required force to cut the occurred connection between the two surfaces tips is less than the force between the metal atoms and that will cause a decrease in wear rate [1, 7]. On the other hand, an increase in applied load will break the oxide film because of its brittleness for its shoots out the friction sliding surfaces for both the discs and specimen during the sliding process, which causes a strong metal contact between them, making the required force to shear its contact tips more than the force between the metal atoms. Also, the effect of pack carburizing quenching contributed to hardness increase. This is obvious in specimens (B) to (E), respectively.

It is difficult to present wear resistance for a time range under 10 minutes. Due to the limited accuracy of the measuring instrument for the adhesive wear test and because the tendency is almost unchanged, thus, only as shown in Fig. 5 served. The disadvantage of this study is that there is a change in the viscosity of the NaCl solution and cane molasses cooling media due to temperature changes, thus affecting the cooling speed.

The development of this research is using a cooling medium of pure cane molasses without being mixed with water and the time between pack carburizing and quenching is shortened.

7. Conclusions

1. Application of 10 % cane molasses cooling medium in pack carburizing quenching treatment caused an increase in the highest carbon content of SS400 low carbon steel.
2. The microstructure of the specimens was pearlite with grain size and grain boundary sized coarse.
3. The surface hardness number of SS400 low carbon steel increased dramatically after the pack carburizing quenching treatment, using the 10 % cane molasses cooling medium causes an increase in the highest carbon content in low carbon steels.
4. The wear resistance increased as a result of changes in carbon content, microstructure, surface hardness number of SS400 steel after treatment, with 10 % cane molasses cooling medium.

Acknowledgments

The authors thank Prof. Rudy Soenoko, Prof. Wahyono Suprapto for invaluable endless collaborations, Prof. I.G.N Wardhana, Director, Postgraduate Doctor of the Mechanical Engineering Program at Brawijaya University for permission to use laboratory and other resource materials.

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