UDC 621
DOI: 10.15587/1729-4061.2021.244118

EFFECT OF PACK CARBURIZING WITH CHICKEN EGG SHELL POWDER AGENT AND VIBRATOR QUENCHING ON THE MECHANICAL PROPERTIES OF AISI 9310 STEEL

Sinarep Sinarep
Master of Tehnical Sciences, Lecturer*
Sujita Darmo
Corresponding author
Doctor of Technical Sciences, Senior Lecturer*
E-mail: sujita@unram.ac.id
*Department of Mechanical Engineering
University of Mataram
Jalan Majapahit, 62, Mataram,
Nusa Tenggara Barat, Indonesia, 83125

The effects of vibrator quenching (QV) on the carbon content, microstructure, and mechanical properties (surface hardness number, wear resistance) in the pack carburizing of AISI 9310 steel were studied. The aim of this research is to increase the surface hardness and improve the wear resistance of AISI 9310 steel. The problem that often occurs in the quenching treatment after pack carburizing is that the thick cooling medium does not evenly wet the surface of the specimen, so that the cooling rate is not uniform, the impact is the distribution of the specimen surface hardness is not the same. Therefore, it is necessary to research the implementation of the vibrator in the quenching treatment.

The specimens were treated with pack carburizing at a temperature of 875 °C, soaking time for 3 hours. The carburizing agent consisted of chicken egg shell powder (CESP) and rice husk charcoal (RHC) with various weight ratios of 5 %:95 %, 15 %:85 %, and 30 %:70 %. Followed by quenching treatment using a 10 % cane molasses cooling medium and vibrator. Hardness testing was carried out using a Vickers microhardness tester, wear resistance test using the pin-on-disc method, and a scanning electron microscope (SEM-EDX) was used to observe changes in the microstructure and carbon elemental content on the specimen surface.

The results showed that the application of VQ caused the formation of a small martensite microstructure while without VQ it was large martensite and a few of residual ferrite. The highest surface hardness number is 685 kg/mm^2, the wear resistance is 0.32 cm/mg for pack carburizing, using carburizing agent 70 % RHC, 30 % CESP and VQ. VQ causes a more even distribution of the thick cane molasses cooling medium so that the cooling rate of the specimens is uniform

Keywords: AISI 9310 steel, vibrator quenching, pack carburizing, surface hardness number, wear resistance

Received date 29.10.2021
Accepted date 03.12.2021
Published date 22.12.2021

How to Cite: *Sinarep, S., Darmo, S. (2021). Effect of pack carburizing with chicken egg shell powder agent and vibrator quenching on the mechanical properties of AISI 9310 steel. Eastern-European Journal of Enterprise Technologies, 6 (12 (114)), 12–19. doi: https://doi.org/10.15587/1729-4061.2021.244118*

1. Introduction

AISI 9310 steel is low carbon steel with a carbon content of 0.10 % and other alloying elements such as nickel, chromium, and molybdenum. AISI 9310 steel is mainly used as gear material in the automotive and aerospace industries, components for small arms, clutch parts, piston pins and track rod pins because it has the advantages of large elongation, low brittleness, deformability, good weldability. However, most of the engineering components of these steels wear out quickly due to their low surface hardness. One of the causes of wear is the low hardness number, friction due to surface contact between elements. Surface hardening techniques are widely used to increase friction resistance so that wear due to the contact force (friction) between surfaces decreases and the service life is longer. The study [1] investigated the contact fatigue resistance of hardened ground alloy steel bars. The specimens were of different aircraft grade alloy steels (AISI 8620, 9310, and 4140) and were hardened using different techniques (carburizing, vacuum carburizing and induction hardening) at different case depths. The fatigue life of the bars was determined using a fatigue testing machine. After testing, the microstructure of the bars was examined using metallographic techniques. It is concluded that there is a relationship between surface hardness, casing depth, and wear resistance of test specimens that experience fatigue due to contact forces between specimen surfaces.

A comparative study of dry shear wear resistance between carbon steel, alloy steel and cast iron was considered [2]. This study used a tribometer (CSM Tribometer) adapted to the pin-on-disc (POD) method. The purpose of this research is to determine the coefficient of friction and wear rate. The specimens used were carbon steel (C25, C45), alloy steel (34CrNiMo6) and cast iron (EN-GJS-400-15). From the research, it was concluded that the coefficient of friction of cast iron is lower, the wear rate of the material is reduced, so that cast iron has better shear wear resistance than carbon steel and alloy steel.

The occurrence of wear on mechanical engineering components causes significant economic losses. The wear resistance of equipment components affected by surface hardness has been described in detail [3]. One of the causes of wear is a contact between surfaces so that friction forces occur. The relative motion in one direction or successive motion under the influence of the load causes the stress on the projection to be large enough to cause plastic deformation and adhesion. The adhesion force will increase if the contact surfaces that rub against each other are clean and there are no obstacles during movement [4].

The wear resistance properties of low carbon steel can be improved by increasing its surface hardness, with pack carburizing treatment, detailed in [5]. Pack carburizing is a heat treatment process used to increase the carbon concentration on the surface of steel specimen, in order to obtain a high hardness and toughness core. One of the methods to achieve these characteristics is pack carburizing using charcoal or mineral coal. In [6], pack carburizing heat treatment on the SAE 1020 mild steel is carried out using rice husk as a carbon source. Pack carburizing is carried out using rice husk charcoal that provides carbon to the surface of the piece of steel. These raw materials are mixed with carbonates ($BaCO_3$, Na_2CO_3, K_2CO_3, $CaCO_3$), which are called energizers and are products that accelerate the carburizing reaction, the carbon potential and the transport rate of carbon from the carburizing medium to the surface of the piece [7].

Pack carburizing treatment of low carbon steel has weaknesses, among others, the thickness of the carburizing layer on the surface of low carbon steel is uneven, causing a very significant difference in surface hardness [8]. So, it is very urgent to conduct research on the use of vibrator quenching so that the carburizing layer formed is thicker and homogeneous, the surface hardness is better and the wear resistance is also increased.

Per capita annual chicken egg consumption in the USA is projected to increase from 242.8 per person in 2010 to 289.9 in 2020 [9]. According to statistics released by the United Nations Food and Agricultural Organization (FAO), the average per capita egg consumption for most countries, computed in kilograms per year (shell weight) has continued to increase over the years. Shells of these eggs constitute an environmental nuisance and end up in dumpsites. A chicken eggshell weighs 5–6 g on average and contains about 85–95 % calcium carbonate ($CaCO_3$), 1.4 % magnesium, and other elements in trace percentages [9, 10].

Therefore, studies are devoted to determining the influence of the distribution of cooling media on the specimen surface on changes in the mechanical properties (surface hardness number, wear resistance), carbon content and microstructure of the specimen. To increase the distribution of the cooling medium, which is concentrated/high viscosity cane molasses was tested using a vibrator during quenching.

2. Literature review and problem statement

Carburizing is a heat treatment process used to increase the carbon concentration on the surface of steel specimen, in order to obtain a high hardness and toughness core. One of the methods to achieve these characteristics is pack carburizing using charcoal or mineral coal. Carburizing heat treatment increases the mechanical and wear resistance. Hardening is achieved when the carburizing layer is quenched to form a martensite structure, which results in better wear resistance, fatigue resistance and toughness. Changes in carbon content on the surface of low carbon steel are caused by the phenomenon of carbon diffusion after pack carburizing followed by quenching treatment. Increasing the carbon content increases the surface hardness number. It has been shown that the application of surface treatment increases the hardness number, wear resistance, lowers the coefficient of friction, and increases corrosion resistance [11].

Based on [12], the surface hardness behavior is shown to be in correlation with the grain refinement process accompanied by strain-induced martensitic transformation and the grain boundary activities involved beginning with a certain grain size. After pack carburizing, quenching was carried out with cooling media such as water, salt solution, SAE 40 oil and 20 % cane molasses. The purpose of quenching is to transform marternsite from a ferrite structure, with a fine grain size. During the quenching treatment, the cooling rate and viscosity of the cooling medium that affect the grain size of the martensite transformation have been described [13].

Quenching treatment using 10 % cane molasses cooling media after pack carburizing caused a significant increase in carbon content, the surface hardness of SS400 low carbon steel [14]. The viscosity of the cooling medium reduces the intensity of quenching. Research [15] increased the quenching intensity of 35CrMoV steel with ultrasonic waves. The cooling quenching medium is vibrated to enter the pores of the specimen, so that the cooling rate occurs uniformly.

Improvement of the surface layer of low carbon steel by ultrasonic nanocrystal surface modification (UNSM) is discussed in detail in [16]. They showed by ultrasonic nanocrystal surface modification as a more effective means of improving surface hardness and decreasing compressive residual stress that the improvement is significantly affected by an increase in energy density during treatment. In [17], pack carburizing heat treatment on the SAE 1020 mild steel is carried out using rice husk as a carbon source. Pack carburizing was carried out at a temperature of 950 °C with a soaking time of 7 hours. It was observed in the microstructural analysis and in the surface hardness that the charcoal, unlike the rice husk, provides better characteristics in terms of greater penetration of carbon in the piece and greater hardness.

Despite the advantages of using ultrasonic waves, ultrasonic nanocrystal surface modification (UNSM) is expensive, has complicated equipment, and high maintenance costs [16]. The application of a vibrator in the quenching process is an easier and simpler process to modify the hardness and wear resistance [18, 19].

The effect of the cooling rate of molasses as a cooling medium for high carbon steels was considered [20]. The molasses solution has the same viscosity as engine oil. The results showed that the surface hardness of high carbon steel is not uniform, without cracking the components so that molasses can be an excellent alternative to be used as a cooling medium, even though the cooling rate is not uniform.

The studies that have been carried out have not examined the effect of vibration (ultrasonic, vibrator) on the quenching cooling medium, which is vibrated by the research specimen, have not used cane molasses cooling media and also have not used alternative carburizing agents.

To date, there are no reports on how to increase the absorption of concentrated cooling media such as cane molasses on the entire surface of the specimen during the quenching process, which are written in the open literature. Therefore, in this study, a vibrator was applied to the quenching process after pack carburizing. The goal is that the increase in surface hardness number occurs uniformly, so that the wear resistance of the specimen (AISI 9310 steel) also increases significantly.

3. The aim and objectives of the study

In this study, vibrator quenching was applied after the pack carburizing of AISI 9310 steel with a carburizing agent in the form of a mixture of rice husk charcoal – chicken egg shell powder.

The objectives of the study that has been carried out are:

– to determine carbon content changes in the carburizing layer on the surface of the specimen;

– to observe changes in microstructure due to vibrator quenching treatment;

– to determine the effect of vibrator quenching and carburizing media on the surface hardness of the specimen;

– to determine changes in the wear resistance of the specimen.

4. Materials and methods of the study

In the present research, AISI 9310 steel was used as the sample. AISI 9310 steel is low alloy steel with the chemical composition outlined in Table 1.

In this study, the specimen was AISI 9310 low carbon steel with the mechanical properties as shown in Table 2. The specimen was cylindrical in shape (diameter of 10 mm, length of 20 mm), referring to the ASTM (E384) hardness test standard and the ASTM (G99-04) wear test standard. Tests are carried out at laboratory room temperature conditions.

Table 1

Chemical composition of AISI 9310 steel

Element	Content, %
Ni	3.00–3.50
Cr	1.00–1.40
Mn	0.45–0.65
Si	0.15–0.30
P	0.025 (max)
S	0.025 (max)
Mo	0.08–0.15
C	0.08–0.13

Table 2

Mechanical properties of AISI 9310 steel

Mechanical properties	Value
Hardness, Vickers	284 kg/m^2
Tensile Strength, Ultimate	910 MPa
Tensile Strength, Yield	570 MPa
Elastic Modulus	200 GPa
Shear Modulus	80 GPa
Elongation	18.08 %
Impact Energy	119 J
Poisson's Ratio	0.29

Before testing, the specimens were treated with pack carburizing followed by quenching, using a vibrator, with a layout as shown in Fig. 1. The AISI 9310 low carbon steel specimen was placed in a carburizing box, which had been filled with a carburizing agent. The carburizing agent consists of chicken egg shell flour (CESP) and rice husk charcoal (RHC) with various weight ratios of 5 %:95 %, 15 %:85 %, and 30 %:70 %. Rice husks are taken from the C4 variety rice plant, which is grown by farmers in West Nusa Tenggara, Indonesia. Charcoal is made of rice husks by burning, then made into powder with a milling machine. To obtain uniform rice husk charcoal powder, sieving was carried out with a particle size of 0.15 mm.

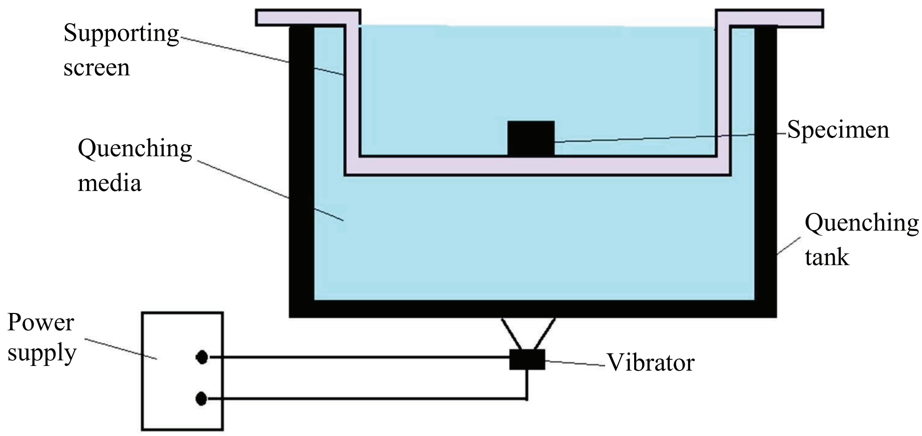

Fig. 1. Basic principle of vibrator quenching

An electric furnace (Carbolite RHF 1700) was used for pack carburizing heating, followed by vibrator quenching (VQ), with a 10 % cane molasses cooling medium. The carburizing agent consists of chicken egg shell powder (CESP) and rice husk charcoal (RHC) with a variation of weight ratios of 5 %:95 %, 15 %:85 %, and 30 %:70 %. The illustration of the basic principle of VQ is shown in Fig. 1. The working principle of vibrator quenching is as follows: in the quenching process, the cooling medium specimen is vibrated, so that the cooling medium wets the specimen surface evenly because the viscous cooling medium needs vibration. The effect of cooling speed on the surface of a homogeneous specimen was studied. After the treatment is completed, tests are carried out including carbon content testing, microstructure observation, surface hardness and wear resistance testing.

Microstructure and carbon content observations were carried out by SEM-EDX (Cam Scan MV2300-Canada) equipped with energy dispersive X-ray (EDX), respectively. The specimens were wet ground in the sequence of 120, 320, 600, and 1000 grit emery paper and polished with 1 μm diamond suspensions, degreased in ethyl alcohol, rinsed in distilled water, and dried with a stream of hot air. They were then immersed into a nital etchant (2 ml of 70 % nitric acid and 48 ml of anhydrous, denatured ethyl alcohol), treated with alcohol swapping, and dried in an air stream. The etched specimens were placed under a microscope on SEM-EDX. So that the measurement of the thickness of the carburizing layer can be carried out. The microscope eyepiece has a measurement scale in micron meters (1/1000 mm) and the total magnification of the microscope used is 400x. Furthermore, the thickness of the carburizing layer was further analyzed to determine the percentage of carbon content.

This Vickers hardness number is calculated by the equation (refer to ASTM E384):

$$HV = \frac{\{2 \cdot Gt \cdot \sin(\alpha/2)\}}{d^2} = 1.854 \cdot \frac{P}{d^2}, \quad (1)$$

where Gt – compressive load (kg); d – average diameter (mm); α – angle of indentor peak=136°.

Testing the wear of the specimen after pack carburizing and vibrator quenching treatment using the pin-on-disc method as shown in Fig. 2 refers to the ASTM G99-04 standard. The specimen is placed on a steel disc with a hardness of 58 HRC and rotated at 940 rpm. So there is friction between the specimen and the steel disc. The wear rate is calculated from the following formula (refer to ASTM G99-04):

$$W_r = \frac{\Delta W}{2\pi nrt}. \quad (2)$$

Explanation of the equation symbols: W_r – wear rate (mg/cm); $2\pi r$ – sliding distance (cm); t – test duration (minutes); n – number of revolutions; $1/W_r$ – wear resistance (cm/mg); $\Delta W = w_1 - w_2$ – weight loss, and n=900 (rpm).

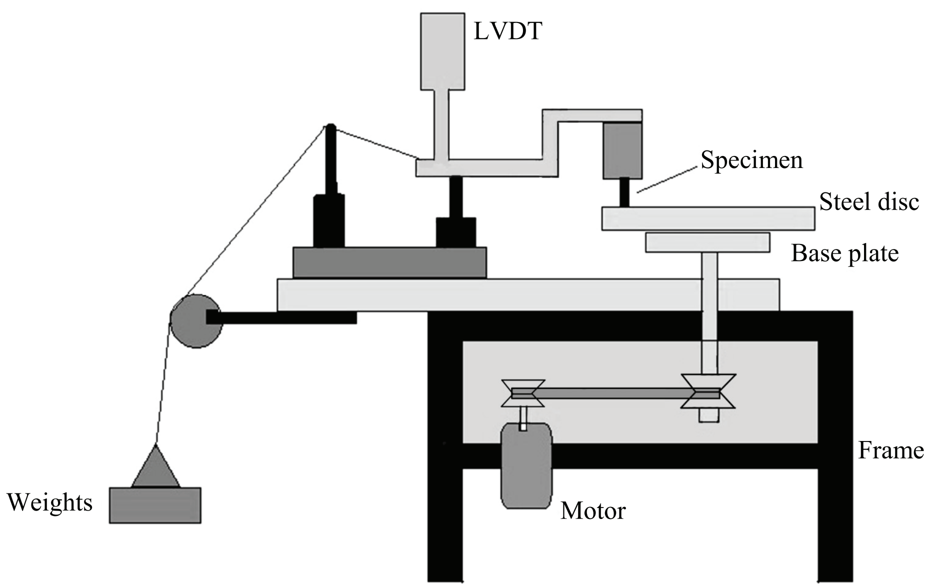

Fig. 2. Pin-on-disc wear testing machine

The computer-controlled pin-on-disc method is used to carry out a wear test according to the ASTM adhesion wear test standard (G99-04) with dimensions of 10 mm in diameter and 15 mm in length. The specimens were placed on stainless steel discs with load variations of 15, 20, 25 and 30 N, different shear speeds of 1.413, 1.884 and 2.356 m/s, disc rotations of 300, 400 and 500 rpm, test duration of 15 minutes and radial shear distance of 90 mm. Direct contact occurs between the specimen surface and the disc by creating roughness on the specimen surface. Discs and samples were cleaned with acetone prior to testing. Before the experiment, the weight of the specimen was measured as w_1 and the weight of the specimen after the wear test was recorded as w_2 with the help of an electronic weighing machine with an accuracy of 0.001 mg. The difference in the weight of the test object before the wear test and after the wear test is called the weight loss (ΔW).

5. Results of the research of vibrator quenching effect on the carbon content, microstructure, and mechanical properties of AISI 9310 steel

5. 1. Carbon content changes in the carburizing layer on the specimen surface

The use of a vibrator in the quenching process affects the carbon content of the specimen. From the analysis of the test results, the carbon content on the specimen surface increased after pack carburizing treatment followed by quenching using vibrator quenching (*VQ*). Significant changes in the carbon content on the specimen surface occurred in the pack carburizing treatment at a temperature of 875 °C, 3 hours of immersion, 30 % CESP, 70 % RHS carburizing agent, quenching with a vibrator, 10 % molasses cooling medium . In general, the carbon content in specimens was affected by the use of *VQ*. The percentage increase in carbon content in the specimens was 0.08 %, 0.12 % and 0.26 %, respectively for no treatment, quenching without *VQ*, quenching using *VQ*.

Table 3 shows that the percentage of carbon content on the specimen surface after treatment increased compared to the initial specimen (0.08 % without treatment). The percentage of carbon content on the surface of the specimen is influenced by the percentage of RHS, CESP in the carburizing agent and *VQ* used in the quenching treatment. The difference in weight percentage of RHC and CESP in the carburizing agent affects carbon diffusion. Because RHC is a carbon source, CESP is a source of $CaCO_3$, which serves to accelerate the diffusion of carbon on the surface of the specimen, according to the results of the study [10]. Due to the vibration of the transducer, the boiling stage of the film is significantly affected, which leads to an increase in the cooling rate. The occurrence of vibration quenching increases the solubility of carbon elements in iron compounds, so that the carbon content on the surface of the specimen increases significantly.

Table 3

Carbon content change in the specimen

Element		Untreated	Without VQ	Using VQ
Ck	Wt %	0.08	0.12	0.26
	At %	20.35	20.35	20.35
Ok	Wt %	18.37	18.33	18.19
	At %	38.86	38.86	38.86
MnK	Wt %	0.43	0.43	0.43
	At %	0.25	0.25	0.25
FeK	Wt %	81.12	81.12	81.12
	At %	40.54	40.54	40.54
Matrix	Wt %	Correction	Correction	Correction
	At %	ZAF	ZAF	ZAF

The research results are in accordance with the statement of research that have been carried out by [18]. The quenching parameters that affect the carbon content of the specimen surface are the cooling rate and the viscosity of the cooling medium [15, 17].

5. 2. Changes in microstructure due to vibrator quenching treatment

Vibrator quenching also affects the formation of the specimen microstructure. Changes in the microstructure of the specimens were observed using SEM-EDX. The test refers to the ASTM E3 microstructure testing standard. Prior to metallographic analysis, the appropriate specimens were etched in 2 % nital solution. Fig. 3 shows SEM-EDX observations of the surface of specimens treated with pack carburizing, cooling at 875 °C, soaking time of 3 hours, compotition of carburizing agent 70 % RHC, 30 % CESP. Followed by quenching with variations using *VQ* and without *VQ*, immersed in 10 % molasses as a cooling medium. Changes in microstructure on the specimen surface after pack carburizing quenching treatment with *VQ* and without *VQ* are shown in Fig. 3, *a*, *b*, respectively. In Fig. 3, *a*, the microstructure is shown, the specimen without *VQ* quenching is still dominated by residual ferrite. The microstructure of ferrite is an interstitial solid solution of carbon atoms in pure iron, so it is commonly called Fe-α. The maximum solubility of carbon in ferrite is 0.025 % at 723 °C, and at room temperature, it has a solubility limit of 0.008 % for carbon. In Fig. 3, *a*, ferrite microstructure (brighter color) changed to pearlite (darker color) and residual ferrite, due to pack carburizing treatment followed by quenching with 10 % cane molasses cooling medium without using a vibrator. By using vibrator quenching, a martensite microstructure is formed, which has a finer grain structure than the ferrite microstructure as shown in Fig. 3, *b*. The different grain sizes in the martensite (darkest color), pearlite and ferrite structures are not only caused by the use of *VQ*, but also due to the different percentages of carburizing agent materials in the pack carburizing treatment.

In Fig. 3, *a*, *b*, the microstructure of the two specimens was quite different. The specimen without *VQ* consisted of pearlite and residual ferrite, while the *VQ* specimen was all small martensite. This is because AISI 9310 steel belongs to low carbon steel. Therefore, if the specimens are wanted to cool from the complete austenite temperature to room temperature without high-temperature transformation, the quenching medium must have a higher quenching intensity. Although the 10 % cane molasses cooling medium had a certain quenching intensity, some local high-temperature changes still existed during the quenching process. When the *VQ* wave was added, the hot steam film on the surface of the specimen was ruptured in advance due to its cavitation and sound effect [19, 21]. Therefore, the microstructure of the *VQ* sample was all small martensite, while the microstructure of the specimen without *VQ* was bulky martensite and a small amount of residual ferrite.

5. 3. Effect of vibrator quenching and carburizing agent on the surface hardness

The vibrator quenching treatment after pack carburizing also resulted in a change in the surface hardness number of the specimen (AISI 9310 steel). The surface hardness testing of the specimen was carried out using the Vickers hardness test method referring to the ASTM E384 standard. The specimen was pressed with a diamond indenter in the shape of a pyramid with a rectangular base and a face angle of 136°. The pressure of the indenter creates a mark on the specimen surface. The test result data is in the form of the diameter of the pressure mark with the indenter, then calculated by equation (1). The results of the calculation of surface hardness to a certain depth are described in a graph, as shown in Fig. 4, *a*, *b*. In Fig. 4, *a*, *b*, the hardness number tends to increase from the surface of the core specimen. The highest hardness number is found on the surface of the specimen. At a distance of 800 m from the surface, the hardness number is constant as the starting material (without treatment), the hardness number is 284 kg/mm^2.

Fig. 4, *a* is the result of testing the hardness of the specimen at a certain depth from the surface. The test was carried out after the pack carburizing treatment at a temperature of 875 °C, 3 hours of soaking time, with carburizing agent materials RHC, CESP and quenched with 10 % molasses sugar cane cooling medium, without VQ. According to Fig. 3, *a*, the surface hardness number of the specimen increases. The distribution of the increasing number of surface hardness starts from the surface (0 μm depth) to 800 μm from the specimen surface. At depths above 800 μm to the core, there is no increase in hardness. Or the hardness number is the same as the initial material (untreated specimen), the hardness number is 284 kg/mm^2. Application of carburizing agent materials (RHC, CESP) with different percentages in the pack carburizing treatment significantly increased the surface

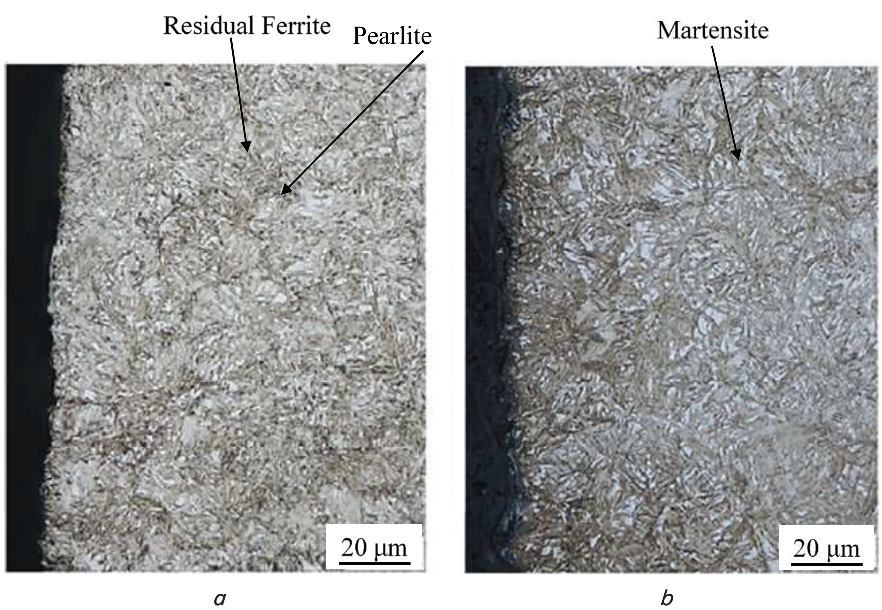

Fig. 3. Surface microstructure of the AISI 9310 steel after pack carburizing quenching (carburizing agent 70 % RHC, 30 % CESP) at 875 °C, soaking time of 3 hours: *a* – without vibrator quenching; *b* – using vibrator quenching

hardness number. They were 425, 450 and 475 kg/mm^2, accordingly, for the percentage of carburizing agent 95 % RHC 5 % CESP:85 % RHC:15 % CESP, and 70 % RHC:30 % CESP. The difference in surface hardness number is caused by the difference in the percentage of CESP, which contains $CaCO_3$ compound, which functions as an energizer (accelerates the diffusion of carbon on the surface of the specimen), so that the carbon content and hardness number also increase.

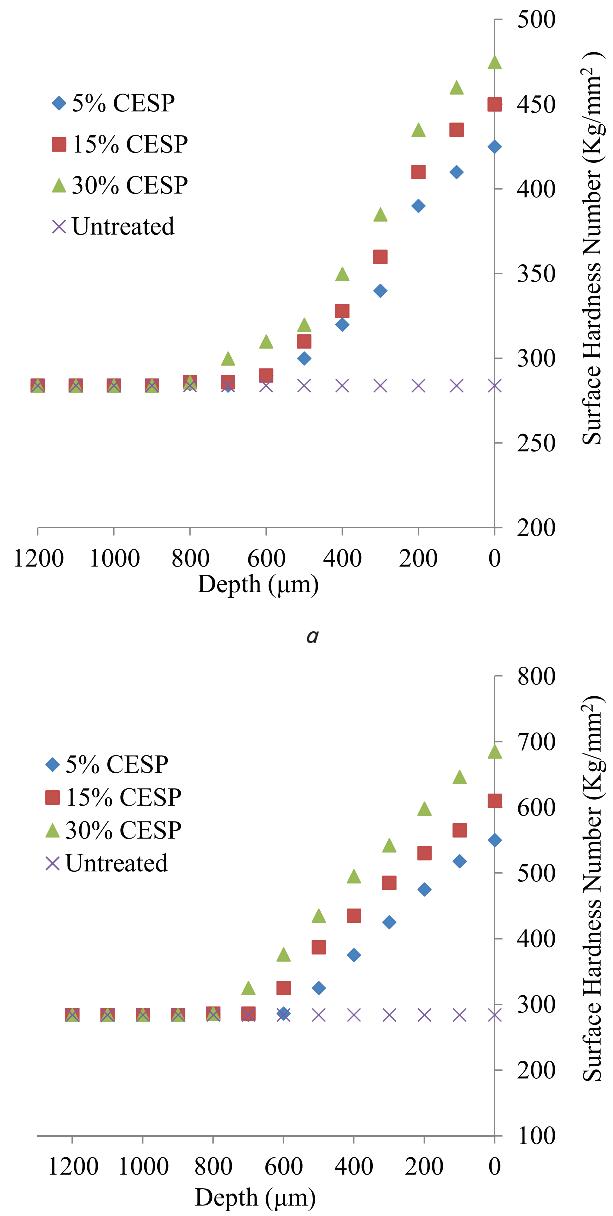

Fig. 4. Effect of carburizing agent on the surface hardness of the specimen: *a* — quenching without vibrator; *b* — quenching with vibrator

Based on the analysis of variance, there is a correlation between the addition of CESP and the use of VQ in quenching on the surface hardness of the specimen as shown in Fig. 4, *b*. The surface hardness numbers were 550, 610 and 685 kg/mm^2, accordingly for 5 %, 15 %, 30 % CESP carburizing agent and using VQ in quenching.

5. 4. Changes in wear resistance after vibrator quenching

The vibrator quenching treatment after pack carburizing also affects the wear resistance of the specimen. The wear resistance test was carried out with the pin-on-disc wear testing machine referring to the ASTM adhesion wear test standard (G99-04). The test results are in the form of weight loss (ΔW), then calculations are carried out based on (2). The result is shown in Fig. 5. Wear testing is carried out on specimens treated by pack carburizing at optimum conditions, i.e. at a temperature of 875 °C, soaking time 3 hours, carburizing agent 70 % wt RHC, 30 % wt CESP, based on Fig. 4, *a*, *b*.

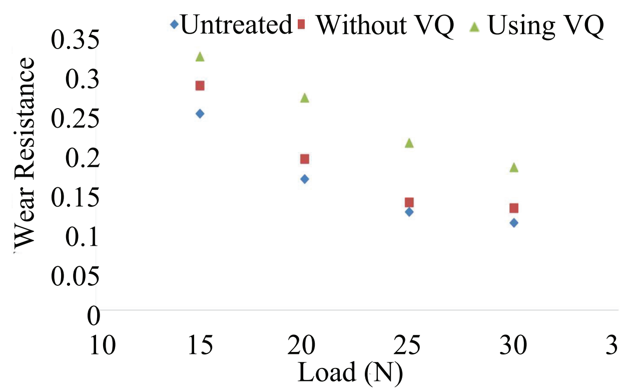

Fig. 5. Effect of vibrator quenching on the wear resistance of the specimen after pack carburizing treatment at optimum conditions

Generally, vibrator quenching (*VQ*) used in pack carburizing treatment has an effect on wear resistance. It is increased compared to untreated specimens. Fig. 5 shows the effect of *VQ* on the wear resistance of specimens after pack carburizing treatment at optimum conditions. It represents the relationship between load and wear resistance. It is shown that pack carburizing treatment with *VQ* gives the highest wear resistance equal to 0.32 cm/mg while the specimen without *VQ* and the untreated one give the lowest wear resistance equal to 0.13 and 0.11 cm/mg, for a load of 30 N.

6. Discussion of the effect of using vibrator quenching after pack carburizing on the mechanical properties of AISI 9310 steel

Pack carburizing treatment and using *VQ* in the quenching process increase the percentage of carbon content on the surface of the specimen as shown in Table 3. The diffusion process causes the percentage of carbon content on the specimen surface to increase. Diffused carbon elements come from rice husk charcoal (RHC) contained in the carburizing agent mixed with chicken egg shell powder (CESP). Rice husk charcoal is a carbon source and CESP is a source of calcium carbonate ($CaCO_3$), which functions as an energizer that accelerates the diffusion process. The rice husk charcoal provides better characteristics in terms of greater penetration of the carbon in the piece and greater hardness [17]. The tissue structure of a chicken egg consists of a protective shell, which contains nearly 68 % calcium carbonate ($CaCO_3$), as discussed in detail in [9, 10]. Diffusion is actually also a chemical reaction between Fe_3C (carbon steel) and $CaCO_3$ at carburizing temperature, as shown in [7].

Fig. 3 shows SEM-EDX observations of the surface of specimens treated with pack carburizing, cooling at a temperature of 875 °C, soaking time of 3 hours, compotition of carburizing agent 70 % RHC, 30 % CESP. Followed by quenching with variations using *VQ* and without *VQ*, immersed in 10 % molasses as a cooling medium. In Fig. 3, *a*, the microstructure is shown, the specimen without *VQ* quenching is still dominated by residual ferrite. The microstructure of ferrite is an interstitial solid solution of carbon atoms in pure iron, so it is commonly called Fe-α. The different grain sizes in the martensite (darkest color), pearlite and ferrite structures are not only caused by the use of *VQ*, but also due to the different percentages of carburizing agent materials in the pack carburizing treatment. Although the 10 % molasses cooling medium has a certain cooling intensity, some local high-temperature changes are still present during the cooling process. When *VQ* vibration is added, the hot vapor layer on the specimen surface breaks first due to cavitation and vibration effects [19, 21]. Martensite is a metastable phase formed when austenite is cooled very rapidly, where in the carbide deposition is suppressed. This occurs when carbon steel or low alloy steel is cooled rapidly [20].

The vibrator quenching treatment after pack carburizing also resulted in a change in the surface hardness number of the specimen (AISI 9310 steel), as shown in Fig. 4. The surface hardness number is affected by carbon content and microstructure, after pack carburizing and quenching processes [17]. Therefore, vibrator quenching (*VQ*) is used in the quenching after pack carburizing treatment, to vibrate the cooling medium (10 % cane molasses). Because the vibration in the quenching tank can increase the absorption of molasses as a cooling medium to the entire surface of the test object. The cooling medium wets the entire surface evenly, due to the vibrator, the cooling speed in the quenching process is evenly distributed on the surface of the specimen.

AISI 9310 is low carbon steel with a percentage of 0.008 % carbon. The wear resistance of the specimen (AISI 9310) steel is influenced by the surface hardness number [4, 13]. Fig. 5 represents the relationship between load and wear resistance. It is shown that using *VQ* in the quenching process after pack carburizing treatment of specimens gives the highest wear resistance while untreated specimens and those without *VQ* give lower wear resistance. The phenomenon shows the effect of the second parameter (*VQ* in the quenching process) and carburizing agent. 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 that causes a thin layer from oxide working as a protective surface film, which limits the contact 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 itself and that will cause a decrease in wear rate [9, 10] for both the discs and the 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 itself.

The advantage of using vibrator quenching is that it is able to stir cane molasses, so that it can be evenly distributed over the entire surface of the specimen. The use of ultrasonic waves in the previous study, which was vibrated by the specimen, was not suitable when using a concentrated cooling medium such as cane molasses in the quenching treatment. It agglomerates, it can't wet the specimen evenly.

It is difficult to even out the distribution of the cooling medium (high-viscosity cane molasses) over the entire surface of the quenched specimen. Cane molasses is difficult to penetrate the pores on the specimen surface, so the cooling process after quenching is not optimal.

The weaknesses of this study are only for cooling media with a maximum concentration of 10 %, carried out at room temperature because cane molasses quickly freezes and evaporates, resulting in lumps and difficulty to stir.

The development of this research is adding a catalyst and increasing the roughness of the specimen, so that the absorption power of the cane molasses cooling medium increases. The use of cane molasses as a cooling medium in the quenching treatment is superior to salt water, oil, causing a pungent odor that interferes with breathing and triggers corrosion of low carbon steel.

The development of this research is using vibrator quenching (*VQ*) to vibrate the cooling medium, so that it is evenly distributed, the cooling process is better and the quality of the quenched specimen is better without reducing the viscosity of the cooling medium.

7. Conclusions

1. The application of vibrator quenching (*VQ*) causes an increase in the percentage of carbon content in the specimen (AISI 9310). The percentage increase in carbon content in the specimens was 0.08 %, 0.12 % and 0.26 %, respectively for no treatment, quenching without *VQ*, quenching using *VQ*.

2. The microstructure of the *VQ* specimens was all small martensite while the microstructure of the specimen without *VQ* consisted of pearlite and residual ferrite.

3. The application of vibrator quenching (*VQ*) after pack carburizing caused an increase in the surface hardness number of AISI 9310 steel dramatically. The hardness number of the specimen before treatment is 284 kg/mm^2. The surface hardness numbers after treatment were 550, 610 and 685 kg/mm^2, accordingly, for 5 %, 30 ,% 15 % CESP carburizing agent and using *VQ* in quenching.

4. The quenching treatment with *VQ* after carburizing pack treatment also causes the wear resistance of the specimen to increase. It is shown that pack carburizing treatment with *VQ* gives the highest wear resistance of 0.32 cm/mg while the specimen without *VQ* and the untreated one give the lowest wear resistance of 0.13 and 0.11 cm/mg, for a load of 30 N.

Acknowledgments

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

References

1. Xie, L., Palmer, D., Otto, F., Wang, Z., Jane Wang, Q. (2014). Effect of Surface Hardening Technique and Case Depth on Rolling Contact Fatigue Behavior of Alloy Steels. Tribology Transactions, 58 (2), 215–224. doi: https://doi.org/10.1080/10402004.2014.960957
2. Nedeloni, L., Korka, Z. I., Pascal, D. T., Kazamer, N., Nedeloni, M. D. (2018). Comparative Study on Dry Sliding Wear Resistance of Carbon Steel, Alloyed Steel and Cast Iron. IOP Conference Series: Materials Science and Engineering, 416, 012026. doi: https://doi.org/10.1088/1757-899x/416/1/012026
3. The Effect of Liquid Nitriding and Carborizing on Adhesive Wear Resistance of Carbon Steel 1020 (2011). Engineering and Technology Journal, 29 (5), 231–240. Aailable at: https://etj.uotechnology.edu.iq/article_30781.html
4. Hawas, M. N. (2013). Effect of Ageing Time on Adhesive Wear of AL Alloy AA6061-T6. Journal of Kerbala University, 11 (4), 145–152. Aailable at: https://www.researchgate.net/publication/320546383_Effect_of_Ageing_Time_on_Adhesive_Wear_of_AL_Alloy_AA6061-T6
5. Singh, S., Singh, D., Sachan, K., Arya, A. (2013). Effect of Soaking Time And Applied Load On Wear Behavior of Carburized Mild Steel. IOSR Journal of Engineering, 03 (02), 10–19. doi: https://doi.org/10.9790/3021-03211019
6. Ihom, A. P., Nyior, G. B., Alabi, O. O., Segun, S., Nor Iv, J., Ogbodo, J. (2012). The Potentials of Waste Organic Materials for Surface Hardness Improvement of Mild Steel. International Journal of Scientific & Engineering Research, 3 (11). Aailable at: https://www.ijser.org/researchpaper/The-Potentials-of-Waste-Organic-Materials-for-Surface-Hardness-Improvement-of-Mild-Steel.pdf
7. García Molleja, J., Milanese, M., Piccoli, M., Moroso, R., Niedbalski, J., Nosei, L. et. al. (2013). 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. Surface and Coatings Technology, 218, 142–151. doi: https://doi.org/10.1016/j.surfcoat.2012.12.043
8. Morita, T., Hirano, Y., Asakura, K., Kumakiri, T., Ikenaga, M., Kagaya, C. (2012). Effects of plasma carburizing and DLC coating on friction-wear characteristics, mechanical properties and fatigue strength of stainless steel. Materials Science and Engineering: A, 558, 349–355. doi: https://doi.org/10.1016/j.msea.2012.08.011
9. Awogbemi, O., Inambao, F., Onuh, E. I. (2020). Modification and characterization of chicken eggshell for possible catalytic applications. Heliyon, 6 (10), e05283. doi: https://doi.org/10.1016/j.heliyon.2020.e05283
10. Arunlertaree, C., Kaewsomboon, W., Kumsopa, A., Pokethitiyook, P., Panyawathanakit, P. (2007). Removal of lead from battery manufacturing wastewater by egg shell. Songklanakarin Journal of Science and Technology, 29 (3), 857–868. Aailable at: https://www.researchgate.net/publication/26469281_Removal_of_lead_from_battery_manufacturing_wastewater_by_egg_shell
11. Wei, Y., Zurecki, Z., Sisson, R. D. (2015). Optimization of processing conditions in plasma activated nitrogen–hydrocarbon carburizing. Surface and Coatings Technology, 272, 190–197. doi: https://doi.org/10.1016/j.surfcoat.2015.04.006
12. Alsultan, S., Quitzke, C., Cheng, Z., Krüger, L., Volkova, O., Wendler, M. (2021). Strain-Induced Martensite Formation and Mechanical Properties of Fe–19Cr–4Ni–3Mn–0.15N–0.15C Austenitic Stainless Steel at Cryogenic Temperature. Steel Research International, 92 (6), 2000611. doi: https://doi.org/10.1002/srin.202000611
13. Nwoke, V. U., Nnuka, E. E., Odo, J. U., Obiorah, S. M. O. (2014). Effect of Process Variables On The Mechanical Properties Of Surface Hardened Mild Steel Quenched In Different Media. International Journal of Scientific & Technology Research, 3 (4), 388–398. Aailable at: https://www.ijstr.org/final-print/apr2014/Effect-Of-Process-Variables-On-The-Mechanical-Properties-Of-Surface-Hardened-Mild-Steel-Quenched-In-Different-Media.pdf
14. 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
15. Jiang, X., Zhou, Y., Shi, C., Mao, D. (2018). Effects of Ultrasonic-Aided Quenching on the Corrosion Resistance of GB 35CrMoV Steel in Seawater Environment. Metals, 8 (2), 104. doi: https://doi.org/10.3390/met8020104
16. Zhao, W., Liu, D., Chiang, R., Qin, H., Zhang, X., Zhang, H. et. al. (2020). Effects of ultrasonic nanocrystal surface modification on the surface integrity, microstructure, and wear resistance of 300M martensitic ultra-high strength steel. Journal of Materials Processing Technology, 285, 116767. doi: https://doi.org/10.1016/j.jmatprotec.2020.116767
17. Llano, J. F., Pérez, E. A., Cárdenas, A. (2019). Husk rice used in the pack carburizing process of the AISI 1020 steel. International Journal of Engineering & Technology, 8 (3), 333–336. Aailable at: https://www.sciencepubco.com/index.php/ijet/article/view/29484
18. Redmann, R., Kessler, O. (2012). Ultrasonic assisted water quenching of aluminium and steel cylinders. International Heat Treatment and Surface Engineering, 6 (3), 115–121. doi: https://doi.org/10.1179/1749514812z.00000000021
19. Barbosa, J., Puga, H. (2017). Ultrasonic melt processing in the low pressure investment casting of Al alloys. Journal of Materials Processing Technology, 244, 150–156. doi: https://doi.org/10.1016/j.jmatprotec.2017.01.031
20. Dodo, M. R., Dauda, E. T., Adamu, M. A. (2016). Investigating the cooling rate of cane molasses as quenching medium for 0.61% C high carbon steels. Metallurgical and Materials Engineering, 22 (1), 39–50. doi: https://doi.org/10.30544/139
21. Komarov, S. (2016). Cavitation Phenomena in Ultrasonic Casting and Their Industrial Application. Tetsu-to-Hagane, 102 (3), 179–185. doi: https://doi.org/10.2355/tetsutohagane.tetsu-2015-097