

В даному дослідженні методом твердого зневуглицювання здійснювалося підвищення пластичності високовуглецевої сталі AISI 420 для поліпшення її механічних властивостей. Зразок поміщали в прямокутну коробку, що містить порошок з черепашок Пінктада максима (ПЧПМ), змішаний з вуглецювальною речовиною з різними процентними варіаціями, і піддавали термообробці в кисневій атмосфері при різних температурах і часі витримки. Результати фазового аналізу показали, що процес твердого зневуглицювання при температурі 900 °C, часі витримки 3 години і додаванні 30 % ПЧПМ до вуглецювальної речовини, що викликає мартенситну мікроструктуру, показник поверхневої твердості і товщина вуглецевого шару зменшилися, а енергія удару високовуглецевої сталі AISI 420 збільшилася. Показник поверхневої твердості, товщина вуглецевого шару, відповідно, зменшилися на 63 % і 60 %, а енергія удару або сила удару збільшилася на 33 %. Дане явище вказує на те, що обробка твердого зневуглицювання викликає дифузію вуглецю від поверхні зразків до вуглецювальної речовини або відбувається зворотна дифузія вуглецю, так як концентрація вуглецю у вуглецювальній речовині вище, ніж на поверхні зразка. Додавання ПЧПМ до вуглецювальної речовини збільшує виникнення дифузії вуглецю з поверхні зразків до вуглецювальної речовини або відбувається зворотна дифузія вуглецю, внаслідок відмінностей у концентрації та впливу ПЧПМ, що містить елементи Са, які діють в якості каталізаторів або активаторів. Результати показали, що процес твердого зневуглицювання з додаванням ПЧПМ до вуглецювальної речовини прискорює дифузію атомів вуглецю з поверхні зразків (процес зворотної дифузії вуглецю), тим самим зменшуючи товщину поверхневого вуглецевого шару, показник поверхневої твердості і збільшуючи енергію удару

Ключові слова: сталь AISI 420, порошок з черепашок Пінктада максима, тверде зневуглицювання, дифузійний процес, показник поверхневої твердості, товщина вуглецевого шару, енергія удару

1. Introduction

Steel AISI 420 is a group of engineering high carbon steel materials which are used in different cutting tools due to specific properties such as good wear and corrosion resistance, high tensile strength, high hardness [1]. High carbon steel has α phase with body centered cubic structure which transforms to γ phase with face centered cubic structure at 910 °C. The high carbon steels contain from 0.60 to 1.00 % C with manganese contents ranging from 0.30 to 0.90 %

[2, 3]. The pearlite has a very fine structure, which makes the steel very hard. Unfortunately this also makes the steel quite brittle and much less ductile than mild steel [4]. This steel is widely used in shear blades, needle valves, surgical equipment, cutlery and industrial equipment due to its high hardness number and tensile strength properties. In spite of these interesting properties, this alloy shows low ductility, brittle and low impact strength [1, 4]. So, its tribological properties should be developed when it is subjected to wear conditions. Those properties can be obtained by surface

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THE INFLUENCE OF THE PACK DECARBURIZING PROCESS WITH PINCTADA MAXIMA SHELL POWDER AGENT ON THE PROPERTIES OF HIGH CARBON STEEL

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modification [5], with carbon diffusion reduces its content on high carbon steel surfaces.

2. Literature review and problem statement

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 tribological and fatigue properties of this steel [4, 6]. The works [7] 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 resistance to corrosion. The enhancement of fatigue wear resistance of gray cast iron by localized laser carburizing is discussed in detail in [8]. They showed that laser carburizing is a more effective means of improving fatigue wear resistance than laser remelting and that the improvement is significantly affected by increase in energy density during treatment. The effect of plasma carburizing and DLC (diamond-like carbon) coating performed on friction-wear characteristics, mechanical properties and fatigue strength of stainless steel is investigated in [9]. 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 the ion carburizing, gas carburizing, plasma carburizing and laser carburizing processes, they are expensive, have complicated equipments and high treatment times and in some situations they can cause formation of deleterious phases and residual stresses [10]. Pack decarburizing is a less expensive and much simpler process to modify the surface of high carbon steel [10, 11]. The influence of oxidation and decarburisation of high carbon chromium steel under charcoal protection during spheroidising is considered in [8]. They showed that the non contact charcoal protection during spheroidising reduces oxidation remarkably, but increases significantly decarburisation compared with annealed in ambient air. In another experiment [12, 13], the effect of different temperature ranges in the temperature range of AC_1 – AC_3 and AC_3 – G ferrite decarburization of silicon spring steel at three different temperatures of heating was investigated. They concluded that the true ferrite decarburized depth follows a parabolic law with the increase of the heating time.

Up to now, no report about pack decarburizing of steel AISI 420 was observed in open literature. Therefore, in this research, the pack decarburizing process was performed on steel AISI 420 to modify the surface by improvement of ductility.

3. The aim and objectives of the study

The aim of the work is to determine the effect of pack decarburizing with an additional Pinctada maxima shell powder (PMSP) in the carburizing agent on the ductility of high carbon steel. This will allow evaluating the effects of

temperature, soaking time and composition of agents during the pack decarburizing process.

To achieve this aim, the following objectives were set:

- to prepare the examined samples (high carbon steel AISI 420) and Pinctada maxima shell powder with a uniform grain size of 150 μm as an additional carburizing agent;
- to determine the surface layer hardness number, thickness of the layer with increased carbon content, impact energy and observation of the microstructure, after the pack decarburizing process with variations of temperature, soaking time and composition of an additional PMSP in the carburizing agent.

4. Material and methods of research

In the present study, high carbon steel AISI 420 with chemical composition of 0.72–0.85 % C, 0.17–0.37 % Si, 0.5–0.8 % Mn, max 0.03 % P, max 0.03 % S, ≤ 0.25 % Cr, 0.1 % Mo, ≤ 0.3 % Ni and ≤ 0.25 % Cu (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 60 % reduction in area. Rectangular box for carburizing box with dimensions of 230×200×123 mm³ was cut from wrought plate.

Charcoal used as a mixture of 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 uniform particle size of 0.15 mm.

Fig. 1 shows a schematic illustration of the pack decarburizing process. Each steel AISI 420 specimen was placed in the carburizing box and covered by the decarburizing agent. It consists of PMSP and charcoal with a weight percentage ratio of 10:90, 20:80, and 30:70 %.

Heated in an electric furnace (Carbolite RHF 1700). The samples were heated at 800 and 900 °C with soaking times of 1, 2, and 3 hours. 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. Finally, the furnace was turned off and allowed the samples to cool to room temperature. Analysis of microstructure, thickness of the carburizing layer, composition of carbon samples was performed by XRD (Unisantix XMD400) and SEM (Cam Scan MV2300) equipped with energy dispersive X-ray (EDX), respectively. The impact strength of samples and ductility of the material were evaluated by measuring the energy impact absorbed by the material with the Impact Testing Machine (JB-W300). Also, a microhardness profile of the cross section of the surface layer was obtained by the Vickers microhardness tester (HV-1000) using 1000 g of penetration load.

Measuring of the carbon layer thickness is done by using a microhardness tester. Test material after etched placed under a microscope on the microstructure of the tester the thickness of the layer will be seen carbon. So that the measurement of the carbon layer thickness can be done because it is inside the eyepiece lens the microscope has a measurement scale in micron meters (1/1000 mm). As for the total magnification of the microscope used is 400 x consisting of 10 x magnifying lens magnification and magnification 40 x objective lens.

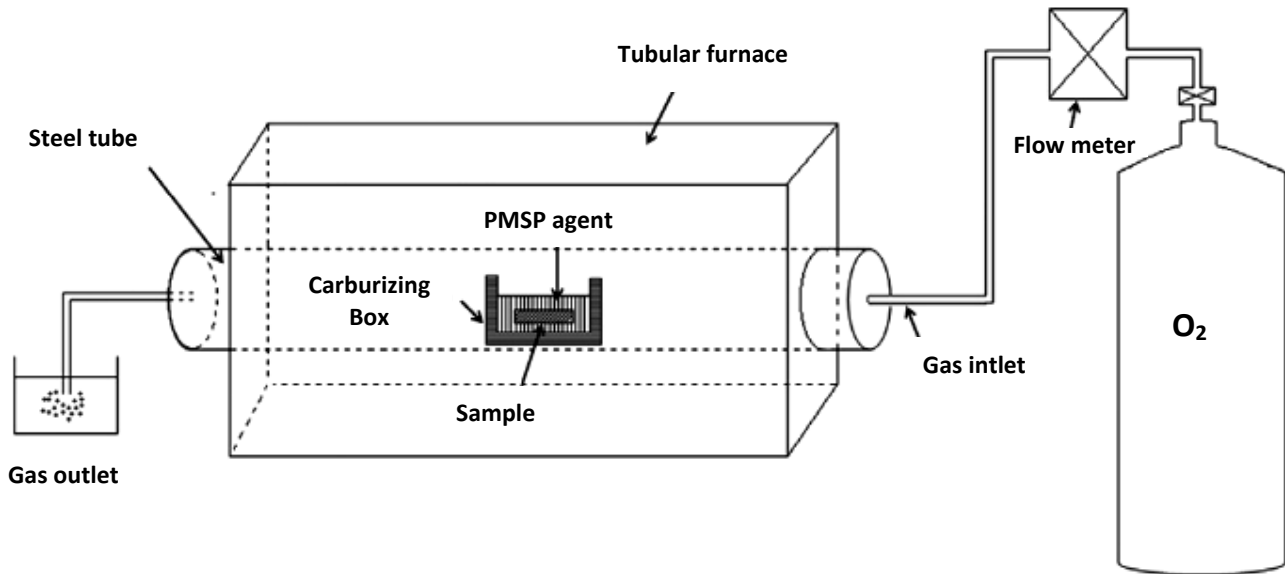


Fig. 1. Schematic illustration of pack decarburizing

4. 1. Determining the composition of carbon content

Based on the composition analysis with SEM-EDX, there was a decrease in carbon composition in the specimens after the pack decarburizing treatment. Significant changes occurred in the pack decarburizing treatment at a temperature of 900 °C, soaking time 3 hours, shown in Table 1.

Table 1

Composition of carbon content of high carbon steel AISI 420 after the pack decarburizing process

Element (wt %)	Composition of Decarburizing Agent (PMSP:Charcoal)		
	10 %:90 %	20 %:80 %	30 %:70 %
C	0.78	0.38	0.198
O	18.93	19.20	19.26
Mn	0.31	0.32	0.43
Fe	79.98	80.10	80.12

The composition of carbon content in the specimens (high carbon steel AISI 420) in general there are changes. In the pack decarburizing process with an additional 10 % PMSP in the decarburizing agent is 0.78 %. It means that an additional 10 % PMSP does not affect the composition of carbon content, because the carbon content of the specimen is 0.72–0.85 %. But the addition of 20 % PMSP and 30 % PMSP causes a decrease in carbon levels to 0.38 % and 0.198 % as shown respectively in Table 1. Compared to the carbon content of the specimen decreased by 8 %, 55 %, and 76 % according to the percentage increase.

Carbon content in the high carbon steel AISI 420 (my specimen) 0.72–0.85 % C, based on material data from suppliers.

Table 1 gives values for the mass content of carbon in the high carbon steel AISI 420 for the pack decarburizing treatment at a temperature of 900 °C, soaking time 3 hours depending on percentage of PMSP addition in the carburizing agent.

With an increase in the percentage of PMSP addition in the decarburizing agent from 10 to 30 %, the carbon content in samples decreases significantly and after 30 % PMSP there is a dramatic decrease in the carbon content, or optimum substance of the decarburizing agent is 30 % PMSP and 70 % charcoal.

The carbon content was 0.198 %, there was 76 % carbon content reduction compared to the carbon content in the specimen. Such a reduction may be due to the fact that an increase in the percentage of PMSP addition in the decarburizing agent improved the process of carbon diffusion from metal surfaces to the decarburizing agent.

4. 2. Observation of the microstructure

Fig. 2 shows SEM – EDX observation of the layer in the sample treated by pack decarburizing at a temperature of 900 °C, soaking time 3 hours and variation of composition of the PMSP agent. The microstructure change on the surface of the specimen after treatment of the pack decarburizing process with variation of the percentage of the carburizing agent 10 % PMSP, 20 % PMSP, 30 % PMSP, each shown as in Fig. 2, a–c.

In the pack decarburizing process, the temperature is 900 °C, soaking time is 3 hours and an additional 10 % PMSP, dependence in Fig. 2, a shows that the microstructure is in the form of martensite, the farther from the core it turns into pearlite and ferrite. Microstructures have not undergone changes such as specimens, high carbon steel. Fig. 2, b, c shows the results of the microstructure of specimens for the pack decarburizing process with the same parameter and an additional 20 % and 30 % PMSP. The microstructure is formed mostly in the form of ferrite and pearlite. The changes of microstructure shape indicate the process of carbon diffusion from the surface specimens to the carburizing agent. The greater the percentage of PMSP addition, increasing the speed of diffusion, so that the microstructure formed on the surface is almost all ferrite. So that the carbon content on the surface of the specimens is less than the specimens that were not treated by the pack decarburizing process.

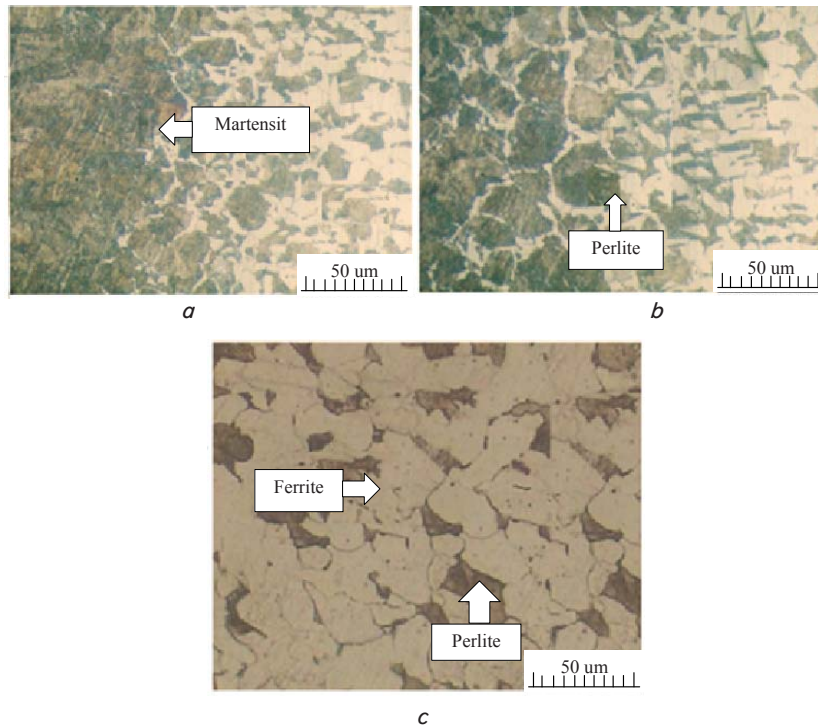


Fig. 2. Microstructure of the surface layer for pack decarburizing at a temperature of 900 °C, soaking time 3 hours, with a variation carburizing agent: *a* – 10 % (PMSP); *b* – 20 % (PMSP); *c* – 30 % (PMSP)

4. 3. Determining the thickness of the layer with increased carbon content

The pack decarburizing process also affects the change in the thickness of the layer with increased carbon content (ICL) on the surface of the specimens, as shown in Fig. 3.

Fig. 3 shows that the pack decarburizing process at 800 °C, the highest reduction in ICL thickness occurs at 3 hours of soaking time and the use of an additional 30 % PMSP in the carburizing agent is 34 % and the lowest is 6 % at 1 hour soaking time, an additional 10 % PMSP in the carburizing agent, and for the pack decarburizing process at a temperature of 900 °C, the highest decrease in ICL thickness occurred at 3 hours of soaking time and the use of an additional 30 % PMSP in the carburizing agent is 37 % and the lowest was 7 % at 1 hour soaking time, an additional 10 % PMSP in the carburizing agent. Depen-

dence in Fig. 3 shows that an increase in the temperature, soaking time and percentage of PMSP addition in the carburizing agent will increase the percentage reduction in the thickness of the ICL. The highest percentage reduction in ICL thickness on the surface of the specimens is 37 % compared to the thickness of the ICL specimen before the pack decarburizing treatment occurred at a temperature of 900 °C, soaking time 3 hours and an additional 30 % PMSP in the carburizing agent. The changes of the thickness of ICL also indicate that the process pack decarburizing caused carbon diffusion from the surface of the specimens to the carburizing agent. So that the thickness of the ICL on the surface of the specimens decreases, proportional to the increase in temperature, soaking time and percentage of an additional PMSP in the carburizing agent.

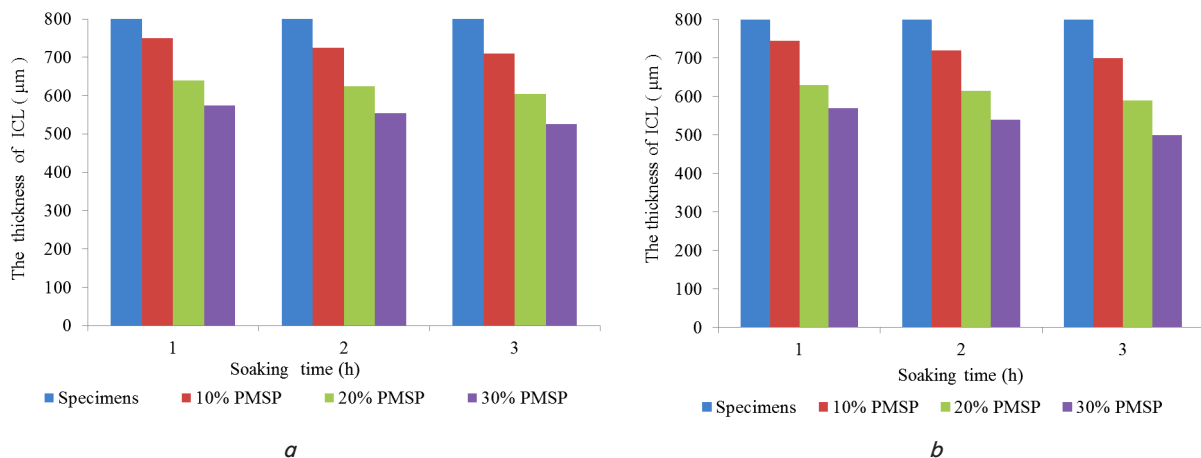


Fig. 3. Thickness of ICL of specimens for pack decarburizing with a variation of an additional PMSP in the decarburizing agent: *a* – at a temperature of 800 °C; *b* – at a temperature of 900 °C

4. 4. Distribution of the surface layer hardness number

We measured the surface hardness number using the method of Vickers hardness number. The basic principle of hardness testing by the Vickers method is to press the specimen with a diamond indenter in the form of a pyramid with a rectangular base and a large face-facing angle 136°. The indenter compressive load causes a trace on the surface of the test object in the form of a curve, shown in Fig. 3.

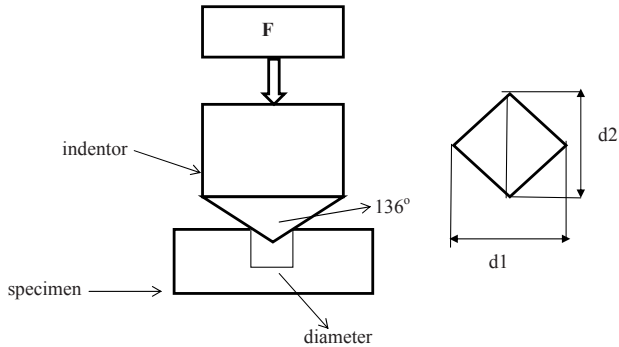


Fig. 4. Trace of the indenter compressive load

This Vickers hardness number is calculated by the formula:

$$HV = \frac{\{2Gt \sin(\alpha / 2)\}}{d^2} = 1,854 \frac{P}{d^2}, \tag{1}$$

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

Fig. 5, 6 is the result of the study of the surface layer hardness number and calculations based on the formula (1).

Fig. 5, *a–c*, shows the results of the distribution of the surface layer hardness number of the core, for pack decarburizing at a temperature of 800 °C, soaking time 1, 2, and 3 hours. After the pack decarburizing treatment, the surface hardness number of specimens decreased. The decrease in the hardness number starts at a distance of 500 μm from the core. The farther away from the core or near the surface the decrease in the hardness number is increasing. At the pack decarburizing temperature of 800 °C, the largest percentage reduction in surface hardness is 59 % compared to the surface hardness of specimens which is 380 Kg·mm⁻², for soaking time 3 hours and an additional 30 % PMSP in the decarburizing agent. The lowest decline of the surface hardness number is 37 %, for soaking time 1 hour and an additional 10 % PMSP in the decarburizing agent, shown in Fig. 5, *a, c*.

Fig. 6, *a–c*, shows the results of the distribution of the surface layer hardness number of the core, for pack decarburizing at a temperature of 900 °C, soaking time 1, 2, and 3 hours. After the pack decarburizing treatment, the surface hardness number of specimens decreased. The decrease in the hardness number starts at a distance of 400 μm from the core.

At a temperature of 900 °C, the reduction in surface hardness numbers is increasing. That is 39 % for soaking time 3 hours and an additional 10 % PMSP in the decarburizing agent and 63 % for soaking time 3 hours and an additional 30 % PMSP in the carburizing agent. This is depicted in Fig. 5, *a–c*, and Fig. 6, *a–c*, showing that the decrease in the hardness number is influenced by temperature, time of soaking and the percentage of addition of PMSP in the decarburizing agent.

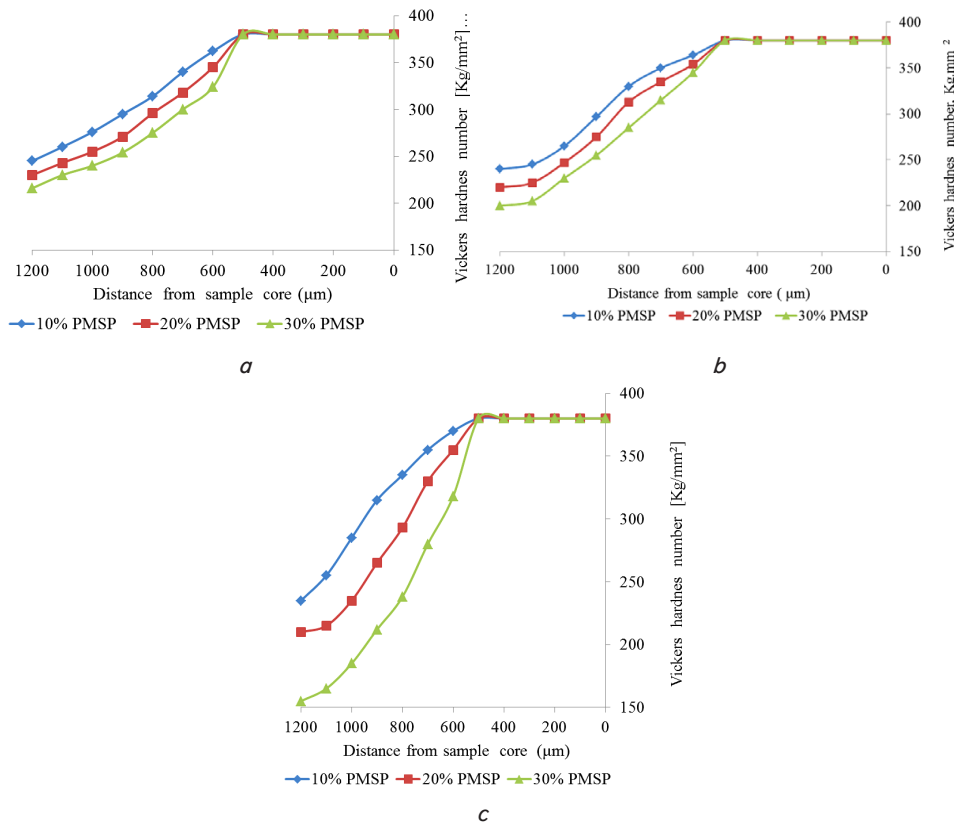


Fig. 5. Hardness number of specimens for pack decarburizing at a temperature of 800 °C with variations of an additional PMSP in the decarburizing agent: *a* – soaking time 1 hour; *b* – soaking time 2 hours; *c* – soaking time 3 hours

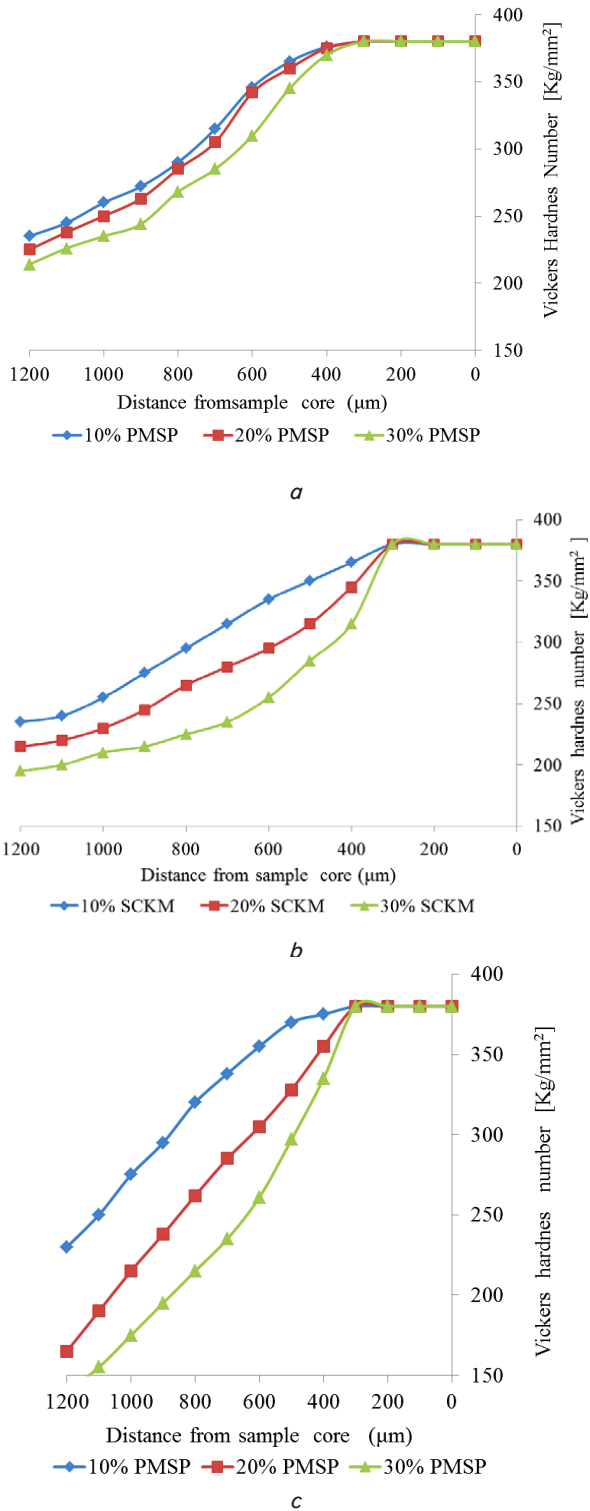


Fig. 6. Hardness number of specimens for pack decarburizing at a temperature of 900 °C with a variation of an additional PMSP in the decarburizing agent: *a* – soaking time 1 hour; *b* – soaking time 2 hours; *c* – soaking time 3 hours

4. 5. Determining the energy impact

Impact testing with the Charpy method is conducted to determine the impact strength and ductility of the material. Fig. 7 shows the dimensions of the specimens, based on the impact testing standard ASTM E 23-56T.

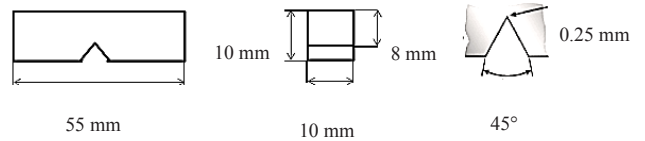


Fig. 7. Dimensions of the specimens based on the impact testing standard ASTM E 23-56T

Impact energy is calculated by the formula:

$$W = G \times \lambda (\cos \beta - \cos \lambda), \tag{2}$$

where W =Energy Impact (J); G =Weight of pendulum (N); λ =Distance of swinging arm (m); $\cos \lambda$ =initial position angle of the pendulum; $\cos \beta$ =final position angle of the pendulum.

The pack decarburizing process causes an increase in energy impact so that the ductility of the specimens also increases. At the pack decarburizing temperature of 800 °C, the largest percentage addition in energy impact is 23 % compared to the energy impact of specimens which is 53 J, soaking time 3 hours and an additional 30 % PMSP agent. The smallest increase in the energy impact is 10 %, soaking time 1 hour and an additional 10 % PMSP agent, shown in Table 3. At a temperature of 900 °C, the addition in energy impact is increasing. That is 33 % soaking time 3 hours and an additional 30 % PMSP in the carburizing agent and 30 % soaking time 3 hours and an additional 30 % PMSP in the carburizing agent and the smallest increase in the energy impact is 25 %, soaking time 1 hour and an additional 10 % PMSP in the decarburizing agent. This is depicted in Fig. 8.

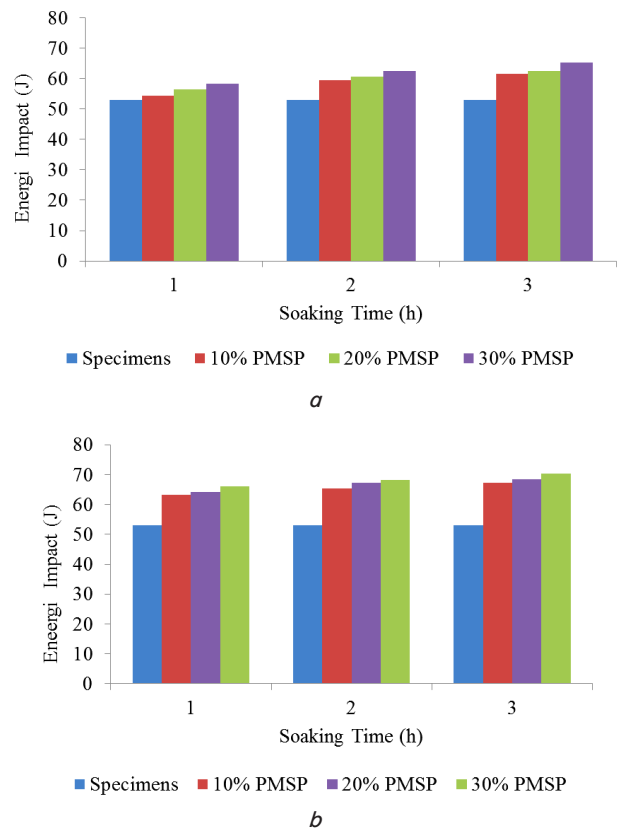
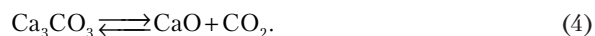


Fig. 8. Energy impact of specimens for pack decarburizing with a variation of an additional PMSP in the decarburizing agent: *a* – at a temperature of 800 °C; *b* – at a temperature of 900 °C

5. Discussion of the effect of the pack decarburizing process with an additional PMSP in the carburizing agent on the properties of high carbon steel

Pinctada maxima is a type of mollusk that has a protective shell, containing Ca carbonate, which is discussed in detail in [14, 15]. In the pack *pinctada maxima* shell powder (PMSP) is added to the carburizing agent or media with different percentage variations. Calcium (Ca) contained in PMSP influences the process of carbon diffusion from the surface of high carbon steel to the carburizing agent. The diffusion process occurs in contrast to conventional carburizing processes, so that called the pack decarburizing process. The result of the pack decarburizing process is the reverse diffusion process, which causes the carbon content, carbon layer thickness and microstructure change, which also results in the hardness number decreasing and ductility increasing. 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:



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



CO gas will change to CO_2 and the reaction will continue like the equation above (equation (3)–(5)).

Based on the phenomenon obtained in this study, there is a process of carbon diffusion from the surface of the specimen to the carburizing agent carbon diffusion media so that the process is commonly called decarburizing, conducted in the study [10–13].

The *Pinctada maxima* shell powder (PMSP), which is considered a pearl shell cultivation waste, was applied as a carburizing agent in the process of pack decarburizing of the high carbon steel. Its advantages are the cheaper price than CaCO_3 or BaCO_3 (energizer carburizing process in

industry), reducing environmental pollution, increasing the economic value of shells that were originally considered to be a waste. Providing opportunities for the prospect of other shells such as *Tegillarca granosa*, *Cerastoderma edule*, *Spisula solidissima* to be used as carburizing agents that function as energizers in the pack decarburizing process so that carbon diffusion is faster.

As shown in Fig. 3, *a*, the thickness of the layer with increased carbon content (ICL) on the surface of the specimens is 625 μm for the pack decarburizing process at a temperature of 800 °C and soaking time 2 hours. Compared with the results of the study [12], which is shown in Table 3, ICL or the surface decarburized depth is 182 μm , at a temperature of 850 °C, soaking time 2 hours and 403 μm for soaking time 8 hours. So the high carbon steel pack decarburizing process with an additional PMSP in the carburizing agent has more advantages.

Reducing the hardness number and increasing the impact energy only on the surface of the specimen to a depth of 800 μm . Its use is only limited to steel material for cutting tools that require ductility on the surface so that it is not easily blunt, but not suitable for steel for construction building.

Heat treatment technology to reduce the surface hardness number by carbon diffusion mechanism, to increase the impact energy or ductility. Modification of the hardness number and ductility, between the surface and core parts is very necessary, especially for steel for cutting tools. The threats are usually the hardness number decreasing of steel material resulting in reduced strength, even though the energy impact or ductility increases.

6. Conclusions

1. The pack decarburizing process at a temperature of 900 °C, for soaking time 3 hours and an additional 30 % PMSP in the carburizing agent causing the martensite microstructure, the surface hardness number, the thickness of carbon layer decreased and the impact energy of high carbon steel AISI 420 increased.

2. The surface hardness number of high carbon steel AISI 420 was 140 $\text{Kg}\cdot\text{mm}^{-2}$ or decreased by 63 %, the thickness carbon layer was 500 μm or decreased by 60 %, and the impact strength was 70.4 J or increased by 33 % after the pack decarburizing process.

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