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Pack-cyaniding: A Comparative Study of Low and High-Temperature Treatment

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Authors' contributions

This work was carried out in collaboration between all authors. Author KJA designed the study, wrote the protocol and wrote the first draft of the manuscript. Authors ALR performed the heat treatment operations and materials characterization of study samples. Authors ARA and MOA supervised the study and interpreted the results. All authors read and approved the final manuscript.

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ABSTRACT

This study made a comparison between low and high-temperature pack cyaniding. Specific focus includes microstructure, surface hardness and case depth of treated samples. Standard specimens machined from mild steel were pack cyanided at 550°C (low temperature) and 950°C (high temperature) using processed cassava leaves with $BaCO_3$ energizer and $BaCl_2$ activator. The heat-treated samples were subjected to micro-examination with a LECO ASTM E384 Microhardness tester, Olympus BH-2 Advanced Optical Microscope and a Cam Scan Series 2 Scanning Electron Microscope. Results from these tests formed the basis for comparison.

Keywords: Clean technology; cassava leaves; pack-cyaniding; microstructure; cyanide; diffusion.

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1. INTRODUCTION

One of the most significant reasons for surface treatment in industries is a modification of engineering properties at the surface of a product. Pack cyaniding is a form of surface treatment which improves the surface hardness of mild steel parts via high or low-temperature heat treatment processes. Cyaniding, as a form of heat treatment, has been reported in all essential details by many authors [1,2-5]. *Pack* cyaniding is a cyaniding technique in which the hardening species(s) is/are sourced from solid materials. The source of the hardening species is of particular interest—a cheap and renewable source is highly desirable for sustainability of the process on an industrial scale [6].

Heat treatment provides a convenient way to modify the service property of a component through an alteration of its microstructure. Factory products—especially cast or machined—are often heat treated to impart desirable engineering properties at the surface and sub-surface. For instance, protection is provided against wear for contacting surfaces by increasing surface hardness through the pack-cyaniding heat treatment.

Pack-cyaniding of mild steel using processed cassava leaves has been successful owing to the presence of cyanogenic glucoside in cassava plant as an accumulation of products of catabolism of amino acids [7]. Consequently, cassava contains some amount of cyanide which could be tapped industrially for case—hardening of steel.

Cassava is rich in waste-to-wealth applications ranging from basic agricultural purposes to specialized industrial functions [8]. A major concern is the toxicity of cyanide to cassava consuming populace and its attendant harmful effects [9]. Current research reports [10,11] have been able to channel this toxic cyanide in the path of industrial utilization with experimental demonstration and analytical modeling of cassava-leaves-enhanced carbonitrided steel.

Cassava contains some amount of cyanide that is often removed as waste during processing for food and sometimes feed. Researchers have devised means of converting this wanton cyanide waste to engineering value via a clean technology pack-cyaniding technique thereby making it of benefit to human use [5,12]. In pack-cyaniding, processed cassava leaves find application as a source of hardening specific carbon and nitrogen [13]. Energizers and activators are used to generally to facilitate extraction of hardening species from Fresh cassava leaves are usually collected, oven-dried, pulverized and subjected to sieve analysis to produce the required particle size. Required particle sizes are mixed with $BaCO_3$ salt by combining 4 volumes of cassava powder with 1 volume of $BaCO_3$ Salt. A firm fireclay luting is provided at the slits between the cyaniding boat and its cover plate. Mild steel sample completely embedded in the cyaniding boat is loaded into a muffle furnace at room temperature. The furnace is heated to 950˚C and held for sufficient time depending on the sample thickness and case depth required. The samples can then be cooled in air or quenched in water/oil as the case may be. The process is the same for low-temperature pack-cyaniding except that the activator is $BaCl_2$ salt and heat treatment temperature is 550°C.

Processed cassava leaves are considered a more sustainable resource for pack-cyaniding considering its relative abundance, ease of processing and low cost. The present study seeks to make a comparison between low and high-temperature pack-cyaniding treatment with specific attention to microstructure, case depth, and hardness.

2. EXPERIMENT

Some AISI 1018 mild steel samples with the chemical composition given in Table 1 were subjected to pack cyaniding heat treatment in pulverized processed cassava leaves of particle size 0.60 µm. The 15 mm long and 10 mm diameter steel samples were treated at 2, 3, 4, and 5 hours in a muffle furnace at 550°C and 950°C. Each pack-cyaniding lot comprised 25% by volume of $BaCO_3$ energizer for high temperature (950°C) treatment and 25% by volume of $BaCl_2$ activator for low temperature (550°C) treatment. Steel samples were embedded in processed cassava leaves inside heat treatment boxes and heated in muffle furnaces from ambient temperature to 950˚C (550˚C for low-temperature treatment). All samples were air-cooled to ambient temperature after heat treatment. Small sections of each sample were mounted in compression mount epoxy media and polished to a final stage of 0.05 µm particle size colloidal silica. All samples prepared for micro- examination were prepared in accordance with ASTM E3-01. Case depths were measured using the Olympus BH-2 Advanced Optical Microscope with an installed Daheng Imavision HV Camera and a calibrated eyepiece. A Cam Scan Series 2 scanning electron microscope was used to reveal the microstructural details of the case and the core at a high magnification of ×3,000 for high temperature samples. The surface hardness was measured using LECO ASTM E384 Microhardness Tester.

Table 1. Chemical composition of AISI 1018 steel

Element	Content, wt%
Carbon	0.15 - 0.20
Silicon	0.15 - 0.30
Manganese	0.60 – 0.90
Phosphorus	0.00 - 0.04
Sulphur	0.00 – 0.05
Iron	balance

3. RESULTS AND DISCUSSION

Results obtained are comparatively discussed in line with the specific focus of microstructure, hardness and case depth.

3.1 Microstructure

Photomicrograph available for the starting mild steel material has a microstructure that is predominantly ferrite and pearlite at ambient temperature (Fig. 1). This material was used for both low and high-temperature pack-cyaniding heat treatment.

In all the samples treated at 950˚C, cases were observed at the edges as a visible region of enhanced diffusion of carbon and nitrogen (Figs. 2-5). Scale measurement was done at representative positions and averages were plotted in Fig. 12. Fine-grained steels are characterized by small grains and numerous grain boundaries. Although carbon tends to be attached to dislocation centers, a large network of grain boundaries may possibly act as constraints inhibiting the drift of carbon into the steel; consequently, diffusing carbon atoms will not travel far before being locked in the subsurface [14]. This is plausible since the grain boundaries can be thought of as regions of discontinuity occurring in the regular repetitive arrangement of atoms.

At high temperatures, both substitutional and interstitial diffusion occur for carbon atoms diffusing into steel [15]. A larger proportion of the diffusion of carbon into steel takes place through the matrix and not through the grain boundary. A large network of grain boundaries is capable of effectively inhibiting movement of dislocation in a material—this inhibition ability results in high toughness [16]. A coarse-grained steel possesses fewer grain boundaries. Movement of dislocation here is less constrained and the diffusion of carbon atoms is enhanced (Samuels, 1999).

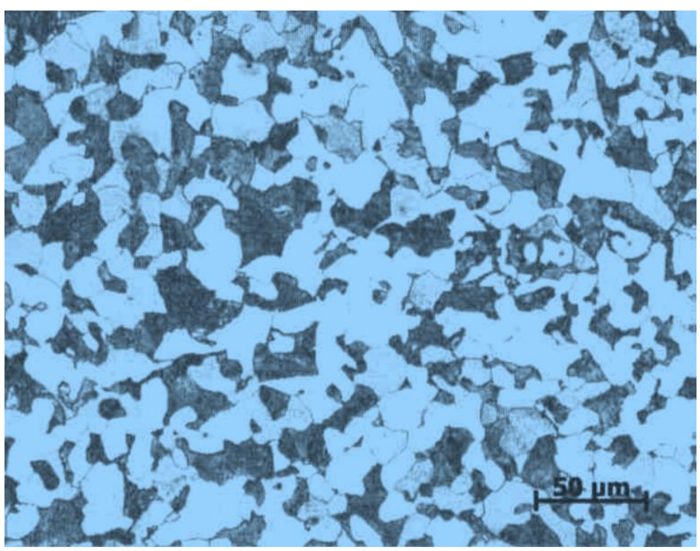

Fig. 1. Photomicrograph of as-received mild steel sample, X200

Fig. 2a. Sample treated at 950˚C for 2 hrs, X200 (Dark upper region represents case)

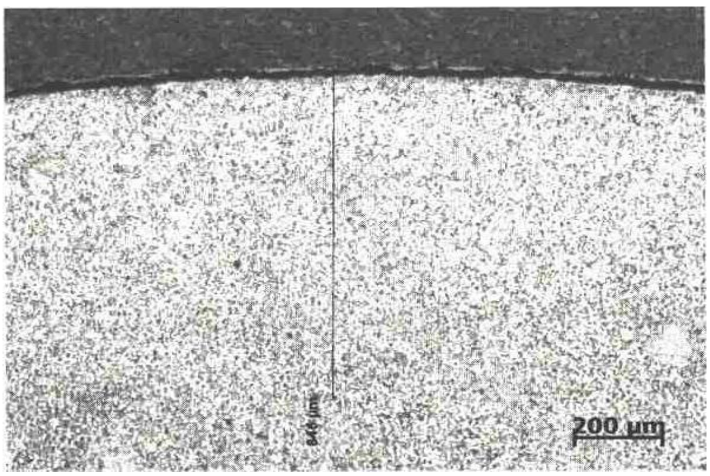

Fig. 2b. Scale measurement on carburized layer (950°C for 2 hrs, X200)
(Scale measurement was done at representative positions and averages were plotted in Fig 12)

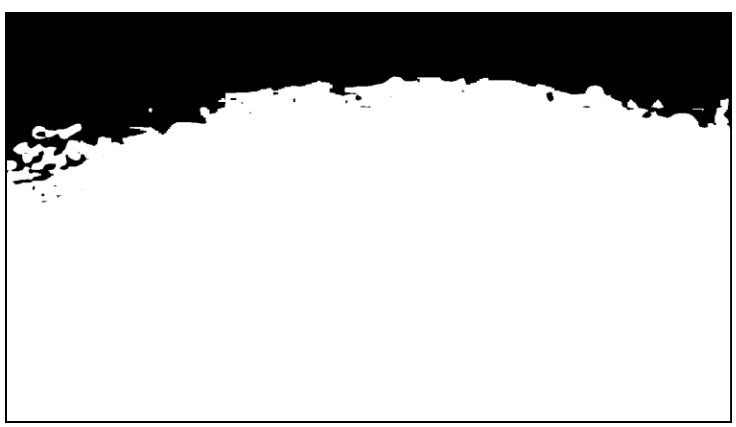

Fig. 2c. Silhouette of scale on carburized layer (950°C for 2 hrs, X200) (Dark upper region represents enhanced hardened case)

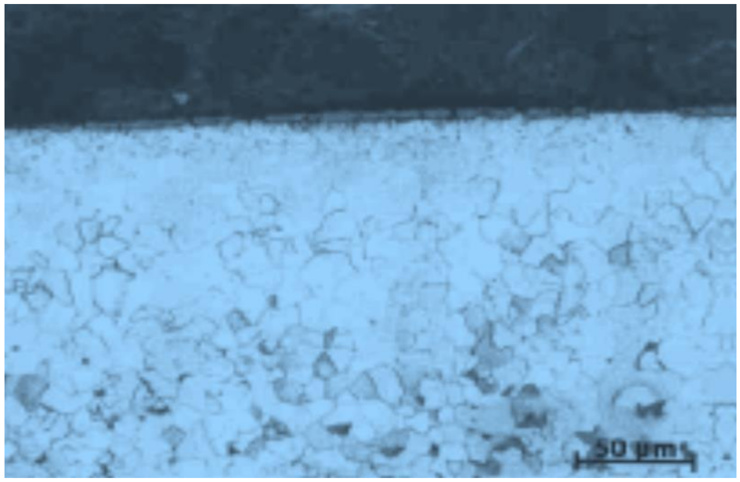

Fig. 3a. Sample treated at 950°C for 3 hrs, X200 (Dark upper region represents case)

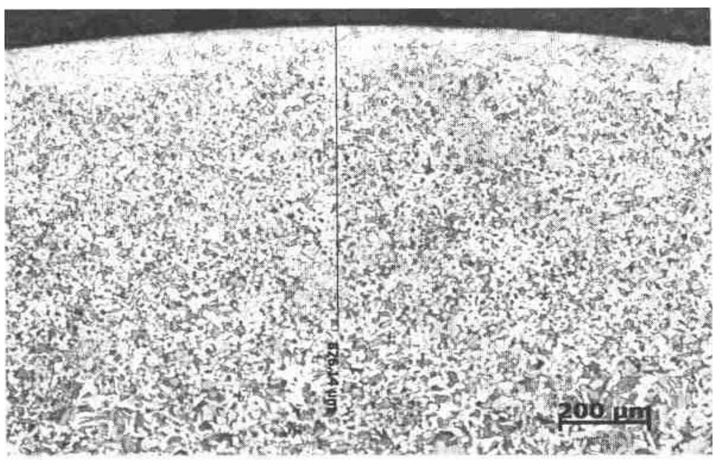

Fig. 3b. Scale measurement on carburized layer (950°C for 3 hrs, X200)
(Scale measurement was done at representative positions and averages were plotted in Fig 12)

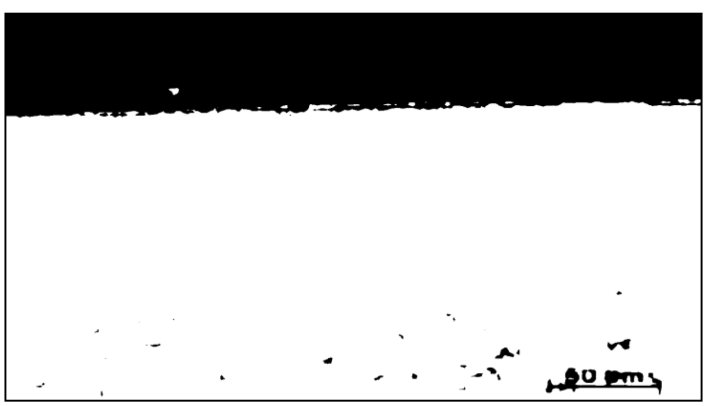

Fig. 3c. Silhouette of scale on carburized layer (950°C for 3 hrs, X200) (Dark upper region represents enhanced hardened case)

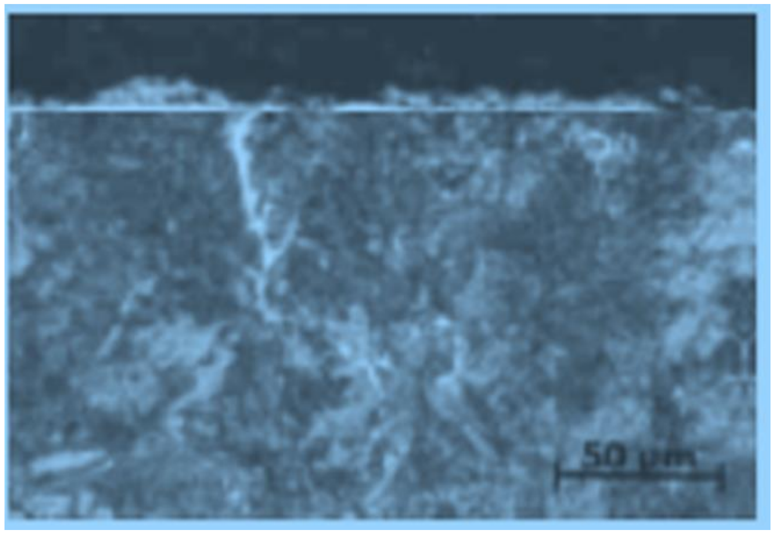

Fig. 4a. Sample treated at 950°C for 4 hrs, X200 (Dark upper region represents case)

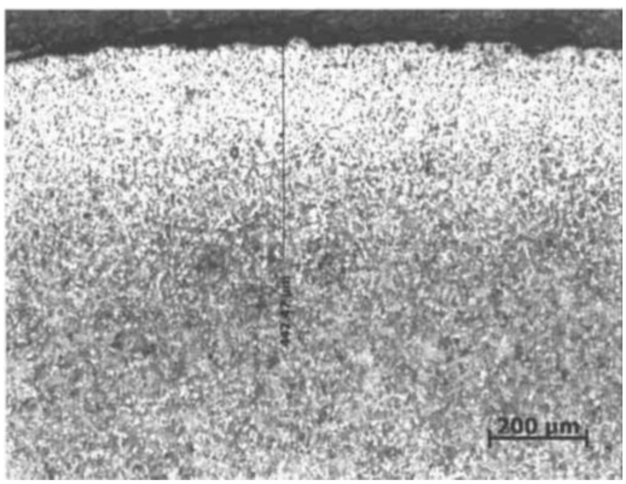

Fig. 4b. Scale measurement on carburized layer (950°C for 4 hrs, X200)
(Scale measurement was done at representative positions and averages were plotted in Fig 12)

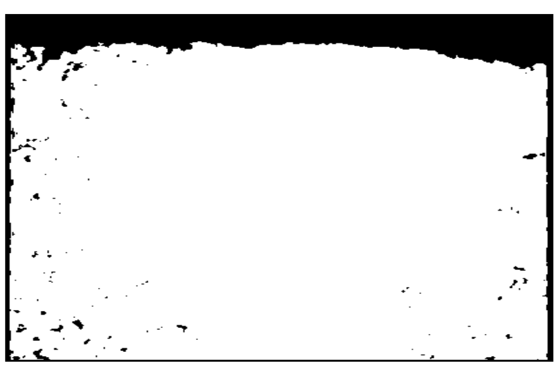

Fig. 4c. Silhouette of scale on carburized layer (950°C for 4 hrs, X200) (Dark upper region represents enhanced hardened case)

The observed microstructure of the cases consists of a predominant pearlite and some ferrite phases while the cores are composed of much less pearlite than ferrite. Scanning electron photomicrographs of these microstructures are presented in Figs. 5-7.

The microstructure of the samples, as revealed by scanning electron microscope, consist of ferrite and pearlite phases. This is indicative of a very good level of toughness and ductility [17].

The micrographs taken at 100X for the mild steel samples pack-cyanided in processed cassava leaves at 550°C lacked visual evidence of a *well-defined* case. Since hardness measurements taken at the surfaces of these samples revealed a consistently higher value at the surfaces of all the samples compared with the cores, it is believed that a hard thin *nitrided* layer was formed at the surfaces through diffusion of atomic nitrogen at 550°C—a temperature value apparently too low for appreciable diffusion of atomic carbon to occur. Compounds/phases which make up this thin nitrided layer are still being studied. At the present stage, however, the microstructure of mild steel heat treated at low-temperature pack-cyaniding of 550°C is predominantly ferrite. Photomicrographs obtained at X100 from optical microscope are presented in the next paragraphs (Figs. 9-11).

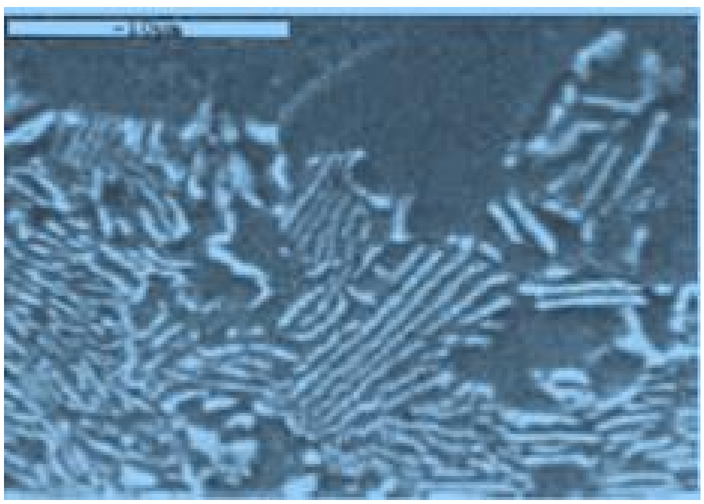

Fig. 5. SEM of case for sample treated at 950˚C for 5 hrs, X3000 (showing appreciable pearlite formation)

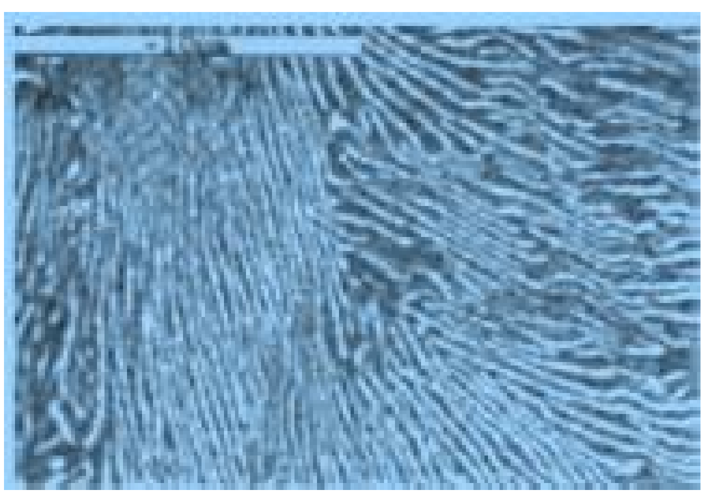

Fig. 6. SEM of case for sample treated at 950˚C for 4 hrs, X3000 (showing very high-density pearlite streaks of lamella arrangement of ferrite and cementite)

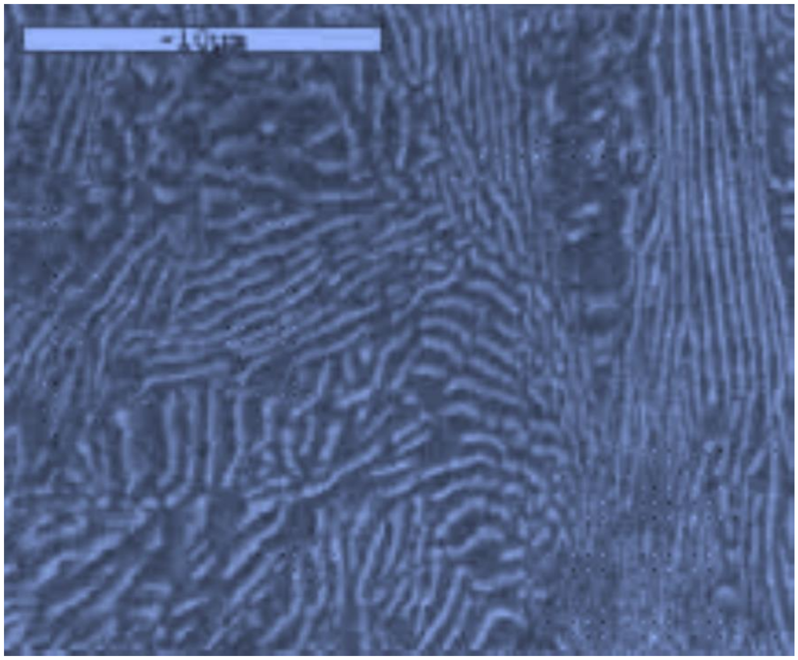

Fig. 7. SEM of case for sample treated at 950°C for 3 hrs, X3000 (showing appreciable pearlite formation)

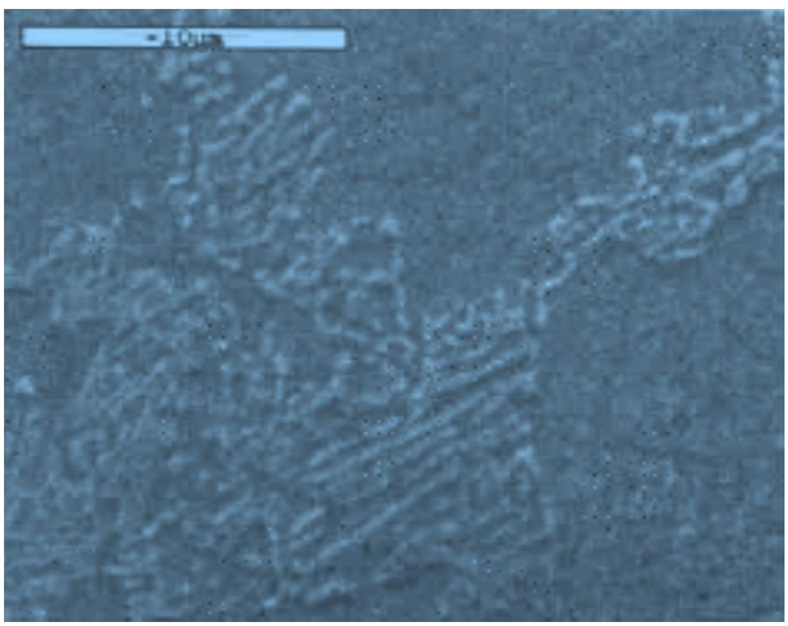

Fig. 8. SEM of case for sample treated at 950°C for 2 hrs, X3000 (showing low density of pearlite in the photomicrograph. Low diffusion time of 2 hrs produced low carbon diffusion hence comparatively low pearlite formation)

3.2 Hardness

Pack cyaniding is essentially a surface-hardening process that involves the thermochemical modification of steel surface and subsurface through thermally activated diffusion of nascent carbon and nitrogen-hardening species. Experimental studies conducted revealed that increased hardness occurred for low and high-temperature samples only at the edges—technically termed *case*. Fig. 12 is a plot of case hardness as a function of treatment time for high and low-temperature pack-cyaniding.

Higher values of Vicker's hardness were obtained for high-temperature treatment compared with low-temperature treatment. The higher hardness is probably owing to longer treatment time and higher temperature. In general, the curves of case hardness as a function of soaking time shown in Fig. 12 increase (with a gentle gradient) with soaking time. A gentle wave characterizes each of the curves. From 3 hours soaking time, the curves rise with a much larger gradient. The reason for this trend is the same as for the curves of case depth versus soaking time. Longer treatment hours allow time for more nascent C liberated from the powder to diffuse into the steel. The higher the number of atoms of C that diffuse the harder is the case formed.

Fig. 9. Sample treated at 550°C for 4 hrs, X100

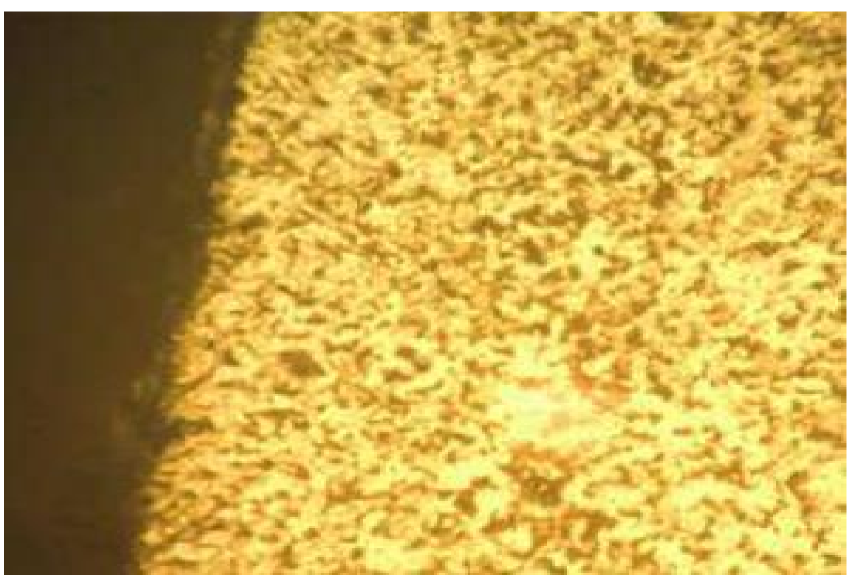

Fig. 10. Sample treated at 550°C for 3 hrs, X100

3.3 Case Depth

The photomicrographs (Figs. 9—11) taken at X100 for the mild steel samples pack-cyanided in processed cassava leaves at 550°C.

Hardness measurements taken at the surface of these samples revealed a consistently higher value at the surfaces of all the samples compared with the cores (130—150 VHN), it is believed that a hard thin *nitrided* layer was formed at the surfaces through diffusion of atomic nitrogen at 550°C—a temperature value apparently too low for appreciable diffusion of atomic carbon to occur. This probably was responsible for a less distinct visual case recognition by the microscopy technique.

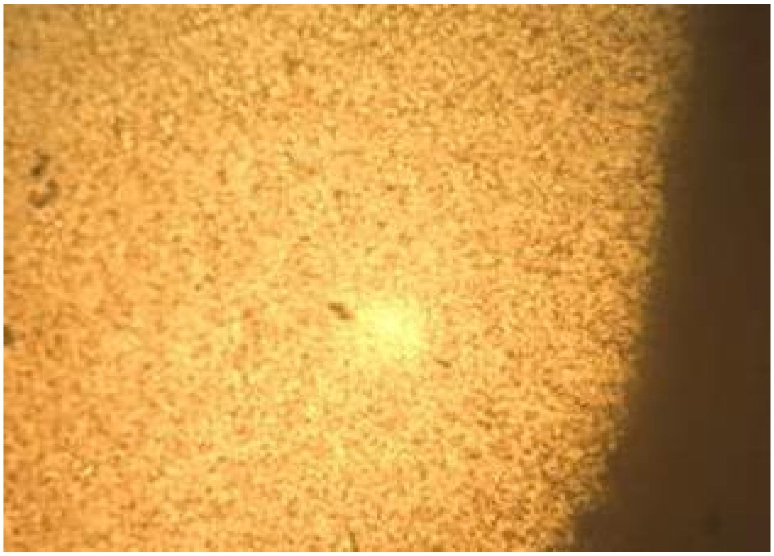

Fig. 11. Sample treated at 550°C for 2 hrs, X100

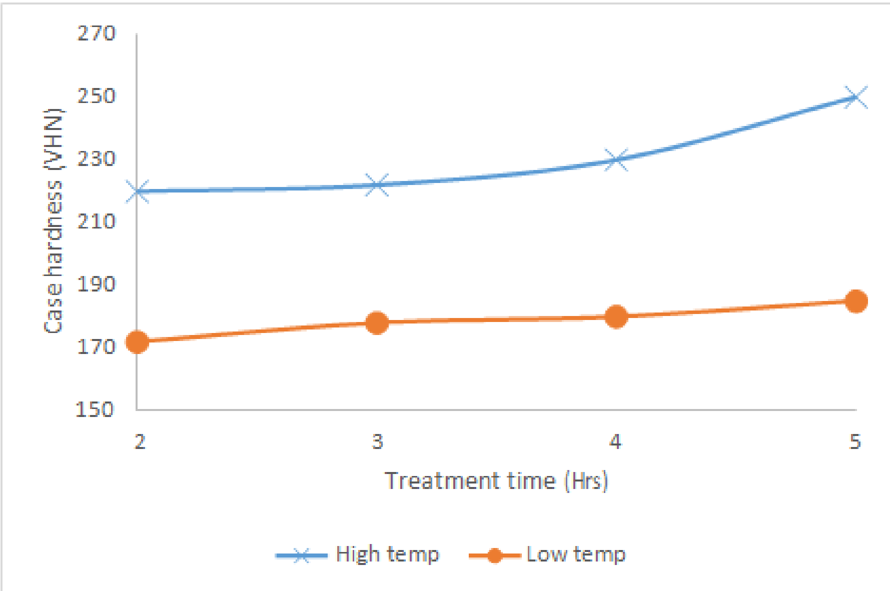

Fig. 12. A plot of case hardness as a function of soaking time

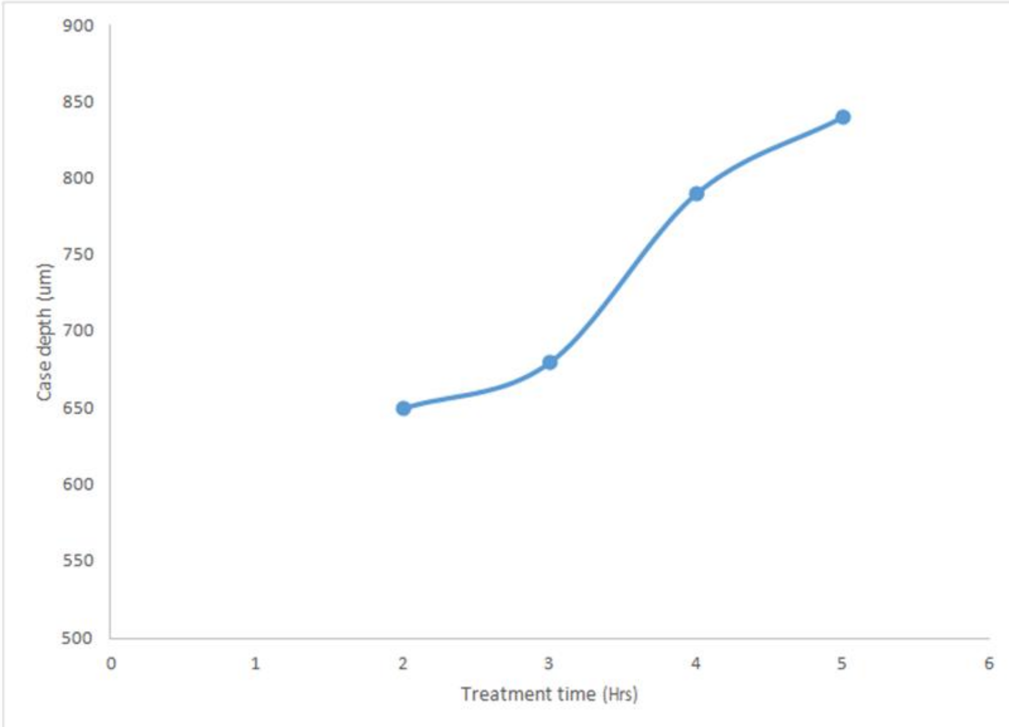

Fig. 13. A plot of case depth as a function of soaking time for high-temperature treatment

For high-temperature pack-cyaniding, the variation of case depth with soaking time is provided in Fig. 13. The curves exhibit a gentle rise for the 0.60 mm processed cassava leaves particle sizes. In general, case depth increases with soaking time. This is because a longer soaking time allows more time for the reaction to proceed. This results in more carbon atoms present in the reaction atmosphere diffusing and migrating deeper into the sample surface. The gentle wavy nature of the curves may be attributed to the impure organic nature of the cassava leaves powder being used as the carbonaceous material for pack cyaniding.

The reason for this gentle wavelike form observed in Fig. 13 may be the impure organic nature of the cassava leaves powder. Conventional salt bath cyaniding produces smooth parabolic curves for lower treatment curves probably because the salts are pure materials.

4. FURTHER WORK

Case hardness profile of samples treated at 980°C is being undertaken. This effort takes notice that hardness profile will be desirable as the work continues. Findings from this experiment will be integrated into the main research work as a corollary to existing results.

5. CONCLUSION

The study confirmed formation of distinct cases after AISI 1018 mild steel samples were pack cyanided at high temperature with characteristically high hardness values and deep cases. However, samples treated at low temperature lacked visual evidence of a well-defined case but they showed improved hardness.

COMPETING INTERESTS

Authors have declared that no competing interests exist.

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