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Microstructural and Case Hardness Studies of Pack Cyanided Low Carbon Steel in Pulverized Cassava Leaves

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Abstract- Investigations were carried out to study the microstructure and case hardness of low carbon steel in pulverized cassava leaves using pack cyaniding technique. This study was conducted to develop milder carburizing compounds that are eco-friendly and sustainable to mitigate the harmful effects of cyanide salts on the environment during deep cyaniding process. The mild steel specimens were examined for both high-temperature diffusion into austenite and low temperature diffusion into ferrite. Results obtained from the microstructural examinations of cyaniding specimens showed that surface hardness of the steel components was substantially increased. Appreciable hard layer cases were formed in specimens pack cyanide at 930^{0}C while only a thin hard layer case was formed at the outer surface of specimens pack cyanided at 570^{0}C. In general excellent hardening properties were obtained with these cassava leaves. It appears that surface hardening of low carbon steel via pack-cyaniding from cassava leaves can be adopted as a heat treatment process for improving its wear performance without polluting the environment and at less cost.

Keywords: Barium carbonate, Cassava leaves, Eco-friendly, Pack-cyaniding, Photomicrograph, Wear characteristic.

1.0 Introduction

The present carburizing compounds in market for surface hardening of mild steel exposed to liquid cyaniding are toxic to the environment and compromises sustainability drives. There is the need to develop milder carburizing compounds that are eco-friendly and sustainable. There are several published data where such liquid cyanide (CN) in molten cyanide salts was applied (Lakhtin Yu, 1979, Rajan et al, 1988 and Janerka et al 2009). Cyaniding is a case hardening process in which both carbon and nitrogen penetrate the surface to a regulated depth (case depth) to raise the hardness and wear resistance of low carbon steel (0.10 to 0.25%C) (Sybil, 1992, Degarmo et al, 1997 and Alagbe, 2012). This type of cyaniding is employed for hardening steel components such as cams, gears, shafts, nut and steering worms.

Surface hardening by carburizing can be classified into three kinds: solid carburizing, liquid carburizing and gas carburizing. In each of these three processes, certain chemicals and methods are used. For the solid carburizing charcoal is used and in liquid carburizing cyanide (CN) is used but in gas carburizing carbon monoxide gas ($C0_2$) is used (Alagbe, 2012). During nitriding process, a white layer of Fe_4N (γ nitride) and Fe_2N (ε nitride) form near the outer layer of steel surface. This layer is problematic as it is very brittle and tends to crack in service (Lakhtin Yu, 1979). Nitrogen has partial solubility in iron. Formation of this layer is caused by high nitrogen potential. In such cases, conditions must be created so as to keep nitrogen potential at the desired level using pulverized cassava leaves. If this is so hard layer case will be formed at the outer surface of samples. The method entails diffusion of nascent cyanide from ripe cassava leaves into the surface of mild steel components via pack cyaniding. The resulting products have much of hardness and wear resistance of the outer steel. Many commercial ‘carburizing’ compounds are available in suitable mixed form (Rajput, 2006). $BaCO_3$ is usually used as the activator. $CaCO_3$ and NaOH are other activators for carburizing process. Many researchers have reported from their studies that $CaCO_3$ can be used in place of other activators such as cow bone to achieve good result (Gerry Egbo et al 2004, Oke and Aderoba, 2000). Cassava contains some amount of cyanide that is often removed as waste during process (Alagbe, 1997). This work intends to convert this wanton cyanide waste to engineering value. The essence of the present study being undertaken is to investigate Microstructural and case hardness of pack-cyanided low carbon steel in pulverized cassava leaves.

2.0 Materials and Methods

The low carbon steel rod used for this work and its chemical analysis was obtained from Universal Steels Limited, Lagos. The experimental work was carried out in the materials laboratory of the Department of Metallurgical Engineering, Kwara State Polytechnic, Ilorin, Nigeria. The mild steel rods was of diameter 10mm and the chemical composition of the steel is C - 0.1968; Si - 0.2422; S - 0.5700; P - 0.0452; Mn - 0.8572; Ni - 0.1231, Cr - 0.1194; Mo -0.0154; V - 0.0034; Cu - 0.2606; W - 0.0016; Ag - 0.0046; Sn - 0.0219, Co - 0.0092, Al - 0.0002; Pb - 0.0003; Ca - 0.0001; Zn - 0.0034; Fe - 98.0419.

Fresh cassava leaves (Manihot Spp.) were collected, oven dried, pulverized and subjected to sieve analysis to produce particle sizes in the range of 0.60mm to 0.20mm. The particle size was divided into: A and B. Group A was mixed with $BaCO_3$ salt by combining 4 volumes of cassava powder with 1 volume of $BaCO_3$ salt and then divided into five portions. This was repeated for group B powder. The cyaniding box consists of a rectangular steel box of dimension 200 x 200 x 40mm. It is opened at one side along the length of the box to allow for packing the steel objects together with the carburizing materials. Mild steel boxes were completely embedded in each member of a batch of group A in a cyaniding box and loaded into a muffle furnace at room temperature. The furnace was heated to 930^0Cand held for 5hours. All the samples were cooled in air. The process was repeated for other four batches but at soaking times of 4, 3, 2 and 1 hour respectively. Group B samples were pack – cyanided in the same way but at 570^0C.

Microstructural examination of as-received and cyanided samples was carried out. Each of the samples was subjected to grinding 240, 320, 400 and 600 grits silicon carbide abrasion paper and polished. The surface of the polished sample was etched in 2% Nital by swabbing the surface with cotton wool soaked in the etchant. The Microstructural examination of the etched surface of the specimens was made under a computerized metallurgical microscope with an in-built camera through which the resulting microstructures were all photographically recorded. Results are shown in Figures 1- 3 Hardness test was conducted on as received specimen while for cyanided specimen; the hardness test was carried out after various case hardening. The LECO micro-hardness testing machine was used to determine the hardness values across the traverse section of the pack cyanided specimens. The results obtained are shown in Figure 4.

3.0 Result and Discussion

Figs. 1a – e represent the photomicrographs of pack cyanided specimens at 930^OC for different soaking times (1-5 hours). Figs. 2a - e show the microstructures of pack-cyanide specimens at 570^0C for different soaking times (1-5 hours). Fig. 3 represents the microstructure of as-received specimen. Fig. 4 shows the case hardness of pack-cyanided specimens with respect to soaking time at 930^0C and 570^0C. In figs. 1a - e, appreciable cases and cores were formed at 930^0C in samples treated. At high temperature the particles size of cassava leaves release their nascent carbon gradually over time allowing time for more carbon atoms to diffuse thereby aid case formation. The longer soaking time allows more time for the reaction to proceed therefore producing cheaper cases. This results in more carbon atoms present in the reaction atmosphere diffusing and migrating deeper into the sample surface. The microstructure of the cases reveals mainly pearlite and some ferrite phases while the cores are composed of much less pearlite than ferrite. In essence, close examination of all microstructures revealed, were ferrite and pearlite phases. In cyaniding at comparative high temperatures, the resistance to wear and hardness is increased and effectively raises the endurance limits of the work. In figs. 2a - e a thin hard layer case was formed at the outer surface of the samples treated at 570^0C. At low temperature, the potential of nitrogen is very high while that of carbon is very low so only nitrogen diffuses appreciably into the steel resulting in nitriding. Beside this, nitrogen iron has greater affinity for nitrogen than carbon in that atomic radius of nitrogen is small relative to that of carbon. In iron-carbon thermal phase diagram, the iron component of mild steel is in the ferritic phase and only nitrogen can diffuse into the steel. By convection, a white layer of Fe_4N (γ nitride) and Fe_2N (ε nitride) forms on the outer layer of surface in nitriding. Nitrogen partially impregnates the steel. It can form a solid solution with ferrite where a composition Fe_4N, is formed at a lesser degree of nitrogen (6%N) or a composition Fe_2N, at a greater degree of nitrogen (8%N) (Forema, 1991). Data of microanalysis of the structure of a cyanided case indicate that appreciable cases were formed in specimens treated at 930^0C while only a thin hard layer case was formed at the outer surface of specimens treated at 570^0C. In fig. 3, no appreciable refinement of the cases was revealed in the microstructures for it was untreated specimen. The curves of case hardness as a function of soaking time shown in Fig. 4 increased with soaking time. The curves have a high gradient from soaking time of 1hour to 3hours. Between 3 hours to 4 hours the curves exhibits a gentle rise. Beyond 4 hours, the curves are almost linear. It was observed that the specimen treated at 930^0C showed a high hardness value while a less hardness value was observed in specimens treated at 570^0C. The curves in the graph revealed that for specimens pack cyanided at 930^0C and 570^0C, a lot of nitrogen diffuses appreciably into the steel at a long soaking time resulting in nitriding. In general, case hardness rises as soaking time increases.

(a) 1 hour

(b) 2 hours

(c) 3 hours

(d) 4 hours

(e) 5 hours

Fig. 1: Micrographs of low carbon steel at different soaking times showing effect at pack cyaniding at 930^0C (x 100)

(a) 1 hour

(b) 2 hours

(c) 3 hours

(d) 4 hours

(e) 5 hours

Fig. 2: Micrographs of low carbon steel at different soaking times showing effect at pack cyaniding at 570^0C (x 100)

Fig. 3: Micrograph of as – received low carbon steel sample (x 100)

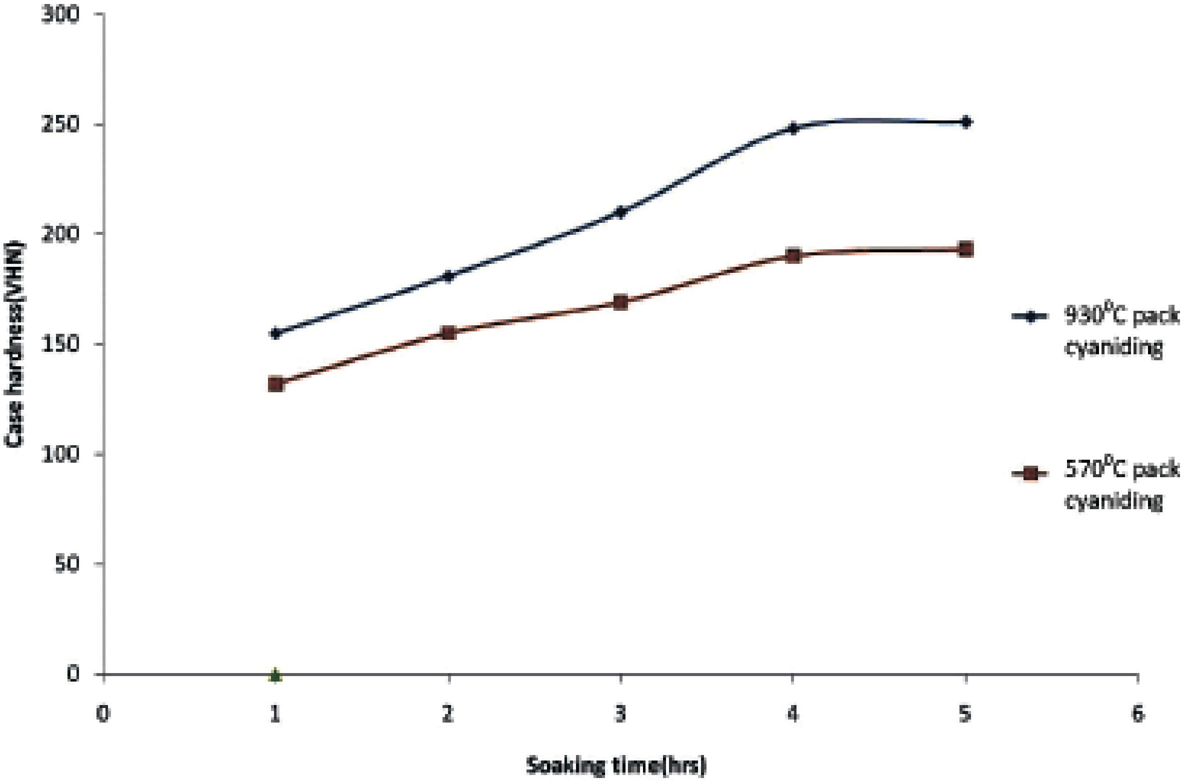

Fig. 4: Case Hardness with respect to soaking time for 570^0C and 930^0C Pack cyaniding

4.0 Conclusions

The following conclusions were drawn from the study

1. Pack – cyaniding is feasible with cassava leaves by the reactions taking place on the metal nitride layer interface where nitrogen has formed a solid solution with ferrous materials.
2. Microanalysis of the structure of a cyanided case indicates that appreciable cases were formed in specimens pack cyanided at 930^0C while only a thin hard layer case was formed at the outer surface of specimens pack cyanided at 570^0C.
3. The cases varied in hardness with cassava leaves as soaking time increases. Specimen pack cyanided at 930^0C is having higher hardness value than specimen pack cyanided at 570^0C.
4. Pack-cyaniding is capable of converting cassava leaves to carburizing compounds without releasing toxic fumes into the air.

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