Case Hardening of Mild Steel Using Animal Bone, Charcoal and Sea Shells as Carburizers

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Abstract
Samples of Mild steel were treated in carburizing media which included animal bone, wood charcoal and sea shells at varied temperatures. Micro structural analyses, chemical composition tests, and mechanical properties tests were carried out on the carburized samples. Results indicated that the treated samples could be used in local production of some engineering components such as gears in place of imported components where hardness is considered together with toughness. The case hardening of the mild steel with charcoal granules gave the highest carburization of 0.905% on the surface with the highest hardness value of 69.3 HRA.
Keywords: Gears, carbon, energizer, carburization, mild steel.

Introduction

Metals are heat treated to obtain better properties for better engineering performances (Higgins, 2010). Different heat treatment methods including case hardening help in micro structural rearrangement of metal atoms which in turn causes controllable changes in the metals properties. Iron melting made of bloomeries produced two layers of metal: one with a very low carbon content that could be worked into wrought iron, and the rest a high carbon cast iron. Since the high carbon iron is hot short, meaning it fractures and crumbles when forged (Craig, 2006); it always needs further treatment. The wrought iron, with nearly no carbon in it is very malleable and ductile, but not hard. Case hardening involves packing the low-carbon iron within a substance with high carbon, then heating this pack to encourage carbon migration into the surface of the iron. This forms a thin surface layer of higher carbon steel, with the carbon content gradually decreasing deeper from the surface. The resulting product combines much of the toughness of a low-carbon steel core with the hardness and wear resistance of the outer high carbon steel as described by Wellyn (1997).

The traditional method of applying the carbon to the surface of the iron involved packing the iron in a mixture of ground bone and charcoal or a combination of leather, hooves, all inside a well-sealed box (Rajan *et al.,* 2001). This carburizing package is heated to a high temperature for a length of time, usually between 900-920˚C, Sanjibku (2009). The longer the package is held at the high temperature, the deeper the carbon will diffuse into the surface. Different depths of hardening are desirable for different purposes: sharp tools need deep hardening to allow grinding and re-sharpening without exposing the soft core, while machine parts like gears might need only shallow hardening for increased wear resistance. The resulting case hardened part may show distinct surface discolourations, the steel darkens significantly and shows a mottled pattern of black, blue and purple, caused by the various compounds formed from impurities in the carbonaceous materials.

This work investigated case hardening of the locally obtained mild steel using charcoal (Ch), sea shells (Ss) and animal bone (Ab) as carburizers. Machines and automobile parts that are used where high impact strength is needed often experience fatigue if not well treated before usage (Zamba and Sumansdi, 2004; Rajput, 2008). With the recent Automobile Policy of the Federal Government, the need for sustained and locally manufactured automobile parts such as axles, gears, cam shafts and crank shafts arises. A good way to solve this problem is through case hardening of mild steel used for the needed parts, whereby, carbon is introduced into the surface of the mild steel in which the inner core of the mild steel remains soft and tough.

In carburization, an amount of carburizer is packed into carburizing box and 20% of Barium trioxocarbonat (V) oxide (Ba_2CO_3) is mixed thoroughly with carburizer in the box. The Ba_2CO_3 acts as an energizer and promotes formation of carbon (IV) oxide (CO_2) gas, which in turn reacts with the excess carbon in the media to produce carbon II oxide (CO). The CO then reacts with the low carbon steel surface to form atomic carbon which diffuses into the steel, as shown in equations 1 -5.

$$BaCO_3\ (s) \rightarrow BaO + CO_2\ (g) \quad (1)$$

$$CO_2\ (g) + C\ (s) \rightarrow 2CO\ (g) \quad (2)$$

Reaction of cementite with carbon monoxide

$$2CO + 3Fe \rightarrow Fe_3C + CO_2 \quad (3)$$

$$CaCO_3 \longrightarrow CaO + CO_2 \quad (4)$$

$$C + CO_2 \longrightarrow 2CO \quad (5)$$

Methodology

The materials used for this work included formulated carburizers (animal bone, wood charcoal, and sea shells), barium carbonate, mild steel of 0.193% C, stainless steel boxes. Mild steel rod of 32 mm diameter was obtained from Universal Steels Limited, Ogba - Lagos and analysed using a spectrometric analyser to obtain the chemical composition of elements present in the steel, Tables 1 and 2. A total of twenty one (21) samples of this steel were then prepared, while the various carburizing media – animal bone, wood charcoal, and sea shells were obtained and pulverized in a ball milling machine into fine powder of size range -90 µm to increase the surface area. Finally, three stainless steel boxes were fabricated to accommodate the carburizing media.

Table 1: Characterization of Carbon Materials

S/N	Carburizers	Percentage Fixed-Carbon (%)	Moisture Content (%)	Ash Content (%)	Volatile Matter (%)
1	Ab	85.61	0.07	8.29	6.10
2	Ch	76.43	0.024	7.20	16.37
3	Ss	78.86	0.058	7.70	13.44

Table 2 Chemical Composition of the Mild Steel before Heat Treatment

C	Mn	Cr	Ni	Mo	V	Si	Fe
0.193	0.7	<0.0001	0.032	<0.0001	<0.0001	0.062	98.92

Sample Preparation

The mild steel rod was machined to tests sizes of 25 mm diameter and 20 mm thickness for hardness and microstructure tests respectively. The surfaces of the samples were all polished into mirror-like surfaces with progressive grinding on grades of silicon carbide (SiC) impregnated emery paper (240-600grits) sizes. Samples were then pre-polished with 1000grit silicon-carbide powder. A rotating cloth pad impregnated with 1µm size alumina polishing powder (APP) was used in polishing with light pressure. Final polishing was carried out using 0.05µm APP suspended in distilled water (Norman, 2007).

Carburization then followed, whereby carburizers were packed into the stainless steel boxes and 20% of Barium trioxocarbonat (V) oxide (Ba_2CO_3) of mass 140g was mixed thoroughly with each medium in each of the boxes. The Ba_2CO_3 acts as an energizer and promotes formation of carbon (IV) oxide (CO_2) gas, which in turn reacts with the excess carbon in the media to produce carbon II oxide (CO). The CO then reacts with the low carbon steel surface to form atomic carbon which diffuses into the steel. Finally, the prepared mild steel samples were buried completely in the pulverized animal bone, wood charcoal or sea shells inside the boxes.

Pack Carburizing Processes

The mild steel samples were buried completely in the carburizing packages, placed in the three stainless steel boxes in the heat treatment muffle furnace at the foundry shop of Federal Institute of Industrial Research Oshodi- FIIRO, Lagos where they were heated and held at 750 ^oC up to 950 ^oC in step of 50 ^oC, as shown in Table 3 and 4. The furnace was allowed to cool before the samples were removed. The carburized samples were then hardened by quenching in water, followed by tempering at 200˚C for one hour. This was done to relieve the internal stresses built up during quenching, and to increase the toughness in the core of the mild steels samples.

Chemical Test

The chemical analysis of the samples was carried out at Universal Steels Limited Ogba, Lagos State, using Spark Test Spectrometry at two different sparks, in order to determine the chemical composition of the steel and to determine specifically, the amount of carbon content present in the steel. This is to classify the steel either as a low-carbon or high carbon-steel. This test showed that the steel contained 0.193 %C, confirming that it is a low carbon steel. After the complete heat treatment of all the samples, the chemical analysis was also carried out at two sparks each, and the results obtained are shown in Table 4.

Table 3: Carburizing Temperature Variation

S/N	Temperature (˚C)	Holding Time (Hours)
1	750	6
2	800	5
3	850	4
4	900	3
5	950	2

Table 4: Carburization Sample Distribution

Time	6 Hours	5 Hours	4 Hours	3 Hours	2 Hours
Temperature (˚C)	750	800	850	900	950
Ab	1	2	3	4	5
Ch	1	2	3	4	5
Ss	1	2	3	4	5

Characterization of the Carburizers

Proximate analysis of the carbon materials for the case hardening process was carried out to know the carbon percentages, the volatile matter, the ash contents and the moisture contents. The results are shown in Table 1.

Hardness Test

The hardness tests were all carried out using Indentec Universal Hardness Tester, with diamond cone (120˚) indentor, Rockwell HRA, minor load 10kg and total load 60kg, in the Metallurgical and Materials Engineering Department of Ahmadu Bello University (ABU) Zaria, at two different indentations. For the untreated As-Received steel sample, 43.7 HRA was obtained, while for the tempered steel samples, the different results obtained were all recorded on a scale of A.

Results and Discussion

The mild steel case hardened with animal bone in the temperature range of 750-850 ^oC increased in hardness up to 55.3 HRA as against the original value of 43.7 HRA, and then decreased to 48.9 HRA at 950 ˚C (Table 5). Also, the case hardened steel using charcoal granules, increased in hardness from 46 – 69.3 HRA at temperatures of 750 – 950 ˚C, and these hardness values lie between the acceptable hardness values of spur gears (50 to 60 HR, ASM standard, 2000.). Case hardened mild steel using sea shells granules, increased in hardness up to 56.5 HRA at 950 ˚C, and then decreased to 39 HRA at 900 ˚C.

The mixture of animal bone and sea shells granules gave hardness value of 44.5 HRA at 950 ˚C, charcoal and sea shells granules gave a hardness value of 44.8 HRA, and finally the three mixtures gave a hardness result of 46.4 HRA. It should be noted that the results of the mixtures hardness value were greatest at the three mixtures, Table 5.

From Table 6, decarburization of the steel samples was experienced at 750^oC in all the three media while a gain in iron was recorded because at this temperature, penetration of atomic carbon into the interstices of the iron atoms had not commenced. Manganese content generally decreased below initial 0.7 % as carbon diffused into the steels. The case hardened steels increased in toughness and wear resistant properties.

Table 5: Hardness Values of Steel Samples after Case Hardening

Temperature (˚C)	Steel Samples	Hardness Results (Hra)
750	Ab	52.1
800	Ab	54.7
850	Ab	55.3
900	Ab	53.5
950	Ab	48.9
750	Ch	46.0
800	Ch	48.4
850	Ch	61.9
900	Ch	58.3
950	Ch	69.3
750	Ss	45.6
800	Ss	40.4
850	Ss	40.9
900	Ss	39.0
950	Ss	56.5
950	50% Ab + 50%Ss	44.5
950	50% Ch + 50% Ss	44.8
950	Ab + Ch + Ss(33.3% each)	46.4

Table 6: Chemical Composition of Steel Samples after Case Hardening

Tempera-ture (˚C)	Carburizer	C	Mn	Cr	Ni	Mo	V	Si	Fe
	Untreated steel	0.193	0.700	<0.0001	0.032	<0.0001	<0.0001	0.062	98.92
750	Ab	0.142	0.697	<0.0001	0.005	<0.0001	<0.0001	0.032	99.038
800	Ab	0.230	0.698	<0.0001	0.013	<0.0001	<0.0001	0.035	98.928
850	Ab	0.211	0.699	<0.0001	0.012	<0.0001	<0.0001	0.036	98.831
900	Ab	0.122	0.701	<0.0001	0.011	<0.0001	<0.0001	0.034	99.039
950	Ab	0.123	0.707	<0.0001	0.009	<0.0001	<0.0001	0.033	99.035
750	Ch	0.013	0.299	0.048	0.018	<0.0001	0.008	0.070	99.443
800	Ch	0.699	0.294	0.044	0.012	<0.0001	<0.0001	0.062	98.75
850	Ch	0.78	0.255	0.045	0.013	<0.0001	<0.0001	0.065	98.70
900	Ch	0.844	0.069	0.044	0.014	<0.0001	<0.0001	0.069	98.662
950	Ch	0.905	0.332	0.039	0.009	<0.0001	<0.0001	0.039	98.572
750	Ss	0.129	0.689	<0.0001	0.012	<0.0001	<0.0001	0.034	99.043
800	Ss	0.028	0.294	0.046	0.007	<0.0001	<0.0001	0.065	99.471
850	Ss	0.349	0.290	0.041	0.001	<0.0001	<0.0001	0.063	99.178
900	Ss	0.006	0.297	0.044	0.002	<0.0001	<0.0001	0.062	99.508
950	Ss	0.118	0.698	<0.0001	0.021	<0.0001	<0.0001	0.041	99.02
950	Ab + Ss	0.065	0.292	0.047	0.029	<0.0001	<0.0001	0.067	99.368
950	Ch + Ss	0.349	0.290	0.041	0.001	<0.0001	<0.0001	0.063	99.178
950	Ab + Ch + Ss	0.241	0.294	0.048	0.012	<0.0001	<0.0001	0.070	99.246

During the carburizing process, carbon was released and subsequently absorbed by the steel samples in different degrees. Highest release and absorption was recorded at 950°C, using charcoal as the carburizing medium. This is in agreement with findings by Panda *et al.*, (2014). Animal bone and sea shells performances were far below charcoal performance as carburizers at all the considered temperatures, except 800˚C and 850˚C for animal bone. Also, at 850˚C, sea shell performed a little bit better than animal bone by raising the carbon level from 0.193 to 0.349. These performances are in agreement with Oyetunji and Adeosun (2012), as well as Fatoba *et al.*, (2013). The mixture of charcoal with sea shell however, gave improved performance as against sea shell alone as a medium at 950˚C. Here, the sea shell acted as an energizer (Fatoba *et al.*, 2013), while the carbon from the charcoal diffused into the steel. The silicon content in the untreated sample was 0.062. The uptake of carbon thereby changed the respective percentages of silicon and iron levels in particular. Manganese content remains relatively the same in samples treated with animal bone. This was however different in samples treated with charcoal. As more carbon diffused into the steel, manganese level reduced automatically, relative to iron content. The implication of this at industrial level is that different combinations of the carburizer/energizers can be predesigned to give desired results on the mild steel for specific industrial application.

Microstructural Analyses

The samples surface microstructures before and after heat treatment are given in the micrographs (Plates 1-7).

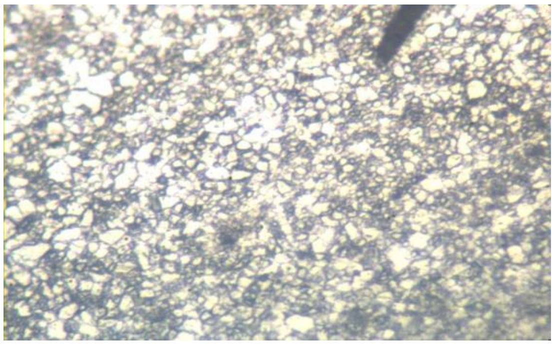

Plate 1: Microstructure of the mild steel before heat treatment × 400; Ferrite (Whitish) and iron carbide (Fe_3C) phases clearly shown in an intermixed assemblage.

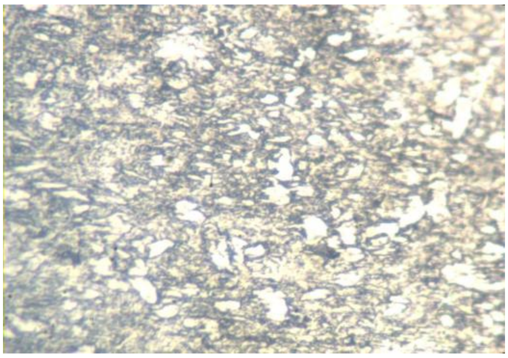

Plate 2: Micro graph after case hardening with animal bone at 950 ^{0}C at magnification of 600, and two hours soaking time. Ferrite is still distinctly seen untransformed.

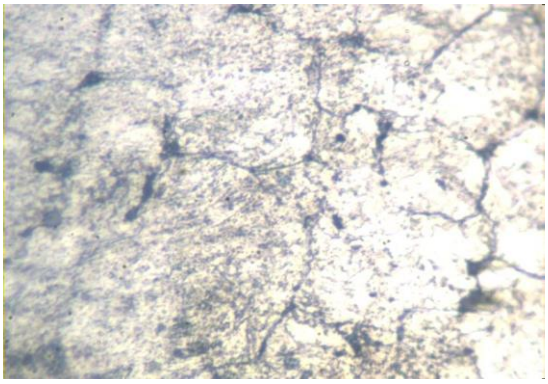

Plate 3: Micro graph after case hardening with charcoal at 900 ^{0}C at magnification of 600, and three hours soaking time. Transformation of Ferrite to iron carbide was favoured by significant uptake of more carbon from the charcoal.

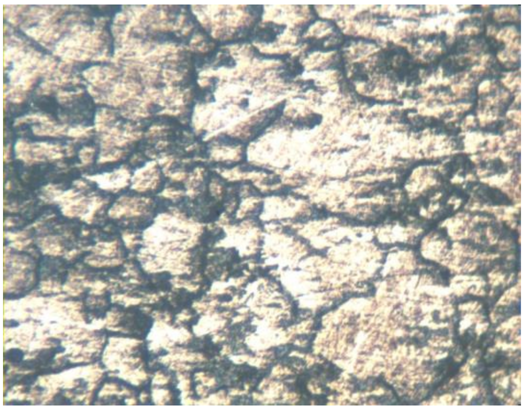

Plate 4: Micro graphs after case hardening with sea shells at 900 ^{0}C ×600, and holding time of three hours. Coarser grains of Ferrite with interstitial iron carbide.

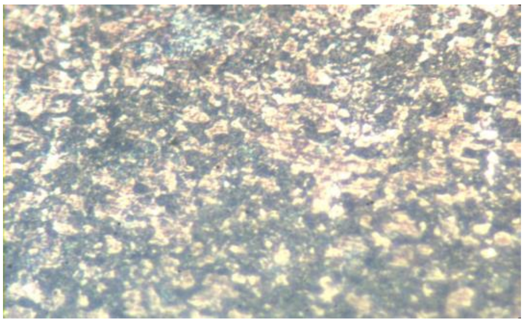

Plate 5: Micro graphs after case hardening with sea shells at 950 ^{0}C at magnification of 600, and holding time of two hours. Ferrite grains with interstitial carbide.

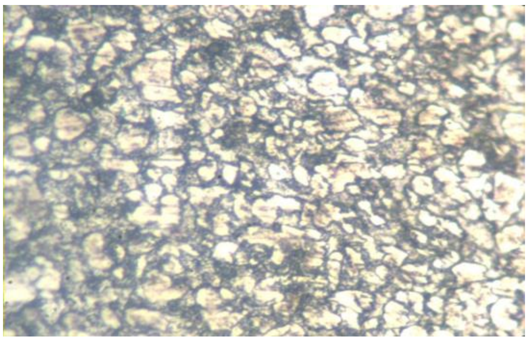

Plate 6: Micrographs after case hardening with the mixture of animal bone, charcoal and sea Shells at 950 ^{0}C at magnification of 600, and two hours soaking time. Finer grains with few transformation from ferrite to iron carbide favoured by carbon uptake.

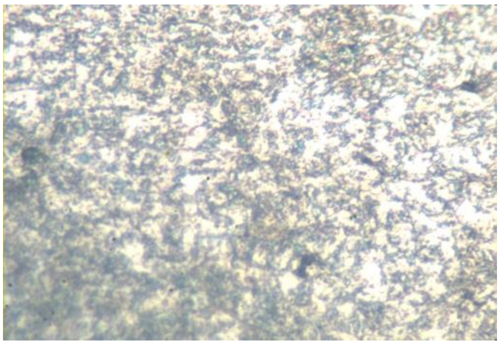

Plate 7: Micro graphs after case hardening with the mixture of charcoal and sea shells at 950 ^{0}C at magnifications of 600, and two hours soaking time. Ferrite transformation also favoured with finer grains.

Microstructure of the steel in Plate 1 clearly shows the hypoeutectic nature of the low carbon steel. Ferrite (whitish) and iron carbide coexisted. Ferrite dominated the field. Very little carbon penetration was recorded with animal-bone case hardening, Ferrite is still conspicuously seen separated from iron carbide as pearlite (Plate 2). Case structure of Plate 3 presents remarkable carburization efficiency. Good phase transition from ferrite in Plate 1, to pearlite was recorded when charcoal was used as the carburizer (Plate 3), this particular sample gave the highest hardness value of 69.3 HRA in Table 6.

The performance of sea shell alone as carburizer was similar to that of animal bone in terms of poor carbon intake; ferrite is still clearly separated from iron carbide as shown in Plate s 4 and 5, this was also responsible for low hardness values of these samples as presented in Table 5. However, there was improvement in carbon intake when charcoal was mixed with each of animal bone and sea shell. Here, both animal bone and sea shell were considered as energizer, while charcoal acted as the real carburizer, Plates 6 and 7. Systematic intake of carbon was therefore recorded, which also led to improved hardness values. Consequently, the work has confirmed the efficacy of using charcoal, animal bone and sea shell in mild steel carburization to achieve desired hardness property.

Conclusions

The case hardened mild steel samples showed significant improvement in their mechanical properties, a proof of effectiveness of the carburizers to induce higher hardness values in the steel samples. These samples could then be utilized in different industrial mild steel applications, like gears as alternatives to imported ones. Highest carbon concentration on the surface of the steel was 0.905% C using charcoal granules, with highest hardness value of 69.3 HRA.

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