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## MICRO-STRUCTURAL AND MECHANICAL CHARACTERIZATION OF PULVERIZED WOOD CHARCOAL AND EGG-SHELL AS ORGANIC CARBON ADDITIVES FOR GREY CAST IRON CARBURIZATION

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### ABSTRACT

The use of wood charcoal as activated carbon for metal carburization is increasing because of its carbon content percentage ranging from 50%–95%. Pulverized carbon from wood charcoal when dispersed into metals offers excellent compression strength which improves the wear characteristics of mild steel material. It is; therefore, important to have the knowledge of the microstructure and mechanical characterisation of activated carbon from wood charcoal for effective and efficient carburization process. In the present study, pulverized wood charcoal and eggshell were characterized using transmission electron microscope (TEM) and x-ray dispersive spectroscopy (XRD) to determine their morphologies and grain size distributions. The characterized organic carbon was further employed to carburize the grey cast iron. It was observed that graphite was deposited on the surfaces of the carburized samples at different temperature compared to as-received sample. This indicates the carbon concentrations. Also, the result of the hardness test revealed that there was increased trend of hardness distribution which is associated with carbon concentration at varying carburizing temperature. Thus, the organic carbons are great substitutes for enhancing the mechanical properties of grey cast iron.

KEYWORD: Wear; Carburization; Grey Cast Iron & Production Technology

Received: Aug 08, 2019; Accepted: Aug 28, 2019; Published: Oct 30, 2019; Paper Id.: IJMPERDDEC201933

### **1. INTRODUCTION**

The environment of application of most engineering components like gears and shaft requires hard and corrosion resistance materials.<sup>1</sup> This is due to their high speed of rotation, vibration, thermal stress and corrosive action of the environment.<sup>2-4</sup> Recently, Orisanmi *et al.*<sup>5</sup> reported that the degradation caused by corrosion is at an increasing trend and measures need to be taken to salvage the problem. Based on this problem, carburization, which is a heat treatment process, has been recognized as a method, which improves the hardness of metals based on the compositions of the carbon additives and the elevation temperature used.<sup>6</sup> According to You *et al.*,<sup>7</sup> carburization as a heat treatment technique has proven effective in reducing processing time and improving the morphologies and mechanical properties of heat treated metals as well as increasing the thickness of surface asperities. The refined microstructure strengthens the modified surface against plastic deformation, which results due to surface defects.<sup>8-11</sup> Wood charcoal has different species and contains pure carbon which made it a potential carbon additive for metal

carburization.<sup>12</sup> It is known to have a fixed percentage of carbon, which increases its heat utilization efficiency.<sup>13</sup> Hussein *et al.*<sup>14</sup> reported that wood charcoal exhibited an amorphous structure; however, undesirable properties, such as low density and Co<sub>2</sub> reactivities, has limited its use in some applications.<sup>15</sup> Therefore, to increase the quality of surfaces of modified metals and general engineering materials, pulverized eggshell has been recognized as a great enhancer as well as excellent corrosion inhibitor.<sup>16-19</sup> According to Sanni *et al.*<sup>20</sup>, eggshell adsorption on stainless steel surface reduces the pitting corrosion effects and improved the strength of the material to function in chloride environment. The adsorption process caused the formation of films of atoms of eggshell which help in improving the mechanical properties of the treated surface.<sup>21</sup> Several additives exist for carburization process such as palm kernel shell, periwinkle shell, cow bones and horns. However, a major drawback to applications of this organic carbon includes processing time and bonding efficiency and most importantly the unfriendly environment of application<sup>22-23</sup> More so, the heterogeneous microstructure of grey cast iron had made the surface modification process difficult. Thus, carburization is an advanced surface treatment method, which can be used to enhance the mechanical properties of metals.<sup>24-25</sup> Further to this manganese, nickel and chromium have been found to exist in the composition of wood charcoal, which necessitates its choice as a carburizing agent.<sup>26</sup>

From study, combination of pulverized wood charcoal and eggshell for surface carburization has not been given due attention. Thus, in the present study, our focus would be on the investigation of pulverized wood charcoal (70 wt.%) and pulverized eggshell (30 wt.%) on the surface carburization of grey cast iron. The hardness property and coefficient of friction behavior would be investigated.

### 2. EXPERIMENTAL PROCEDURE

The pulverized wood charcoal and eggshell were obtained and filtered to 75  $\mu$ m for easy diffusion of the carbon particles into the surface of the metal. Weight fraction of 70 wt.% of wood charcoal and 30 wt.% of eggshell was employed. Grey cast iron samples (HT250) of dimension (30 mm × 20 mm × 5 mm) were obtained and polished with different grades of silicon carbide abrasives to achieve a smooth and flat surface. The substrate material (grey cast iron) was etched in 50 ml of Nital to reveal the variations at the surface. The microstructure and chemical composition of the as-received grey cast iron samples is shown in Figure 1(a) and Table 1 respectively. In order to reduce the effects of moisture on the carburization process, the carburizing agents (pulverized wood charcoal and eggshell) were kept in a vacuum for 3 h at 80°C. Figure 1 showed the microstructural image of the as-received grey cast iron sample. Transmission electron microscope images (TEM) of both the pulverized wood charcoal and eggshell are provided on Figure 2. Similarly, the xray diffractograph of the carburizers are shown in figure 3.

The samples were embedded in the mixture of wood charcoal and eggshell contained in a stainless steel container and loaded into a muffle furnace of 1200°C capacity. Carburization was carried out at 700°C, 800°C and 900°C for three hours, respectively. The micro hardness was measured using a Digital Vickers Micro Hardness Tester using a load of 500 N and dwell time of 15 seconds.

### 3. RESULTS AND DISCUSSIONS

Figure 1 represents the SEM morphology of the as-received grey cast iron, while Figure 2 (a–b) showed the TEM images of both the pulverized wood charcoal and eggshell. The bright regions indicate carbon presence. From Figure 2, it could be observed that both morphologies of pulverized wood charcoal and eggshell were characterized by crystalline structure with small aggregates while some particles are spherical in shape. These size distribution properties indicate their excellent

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diffusion into the substrate (grey cast iron) material during the carburization process. Also, the x-ray diffraction spectra (Figure 3) for the pulverized wood charcoal and eggshell indicate a broad peak at 30°. The similarities in their peaks as observed showed the presence of amorphous carbon in the material. Further to this, smaller peaks at 28° and 40° for the wood charcoal also indicate the presence of graphite.



Figure 1: SEM Morphology of As-Received Grey Cast Iron.



Figure 2: (a)TEM Image of Pulverized Wood Charcoal. (b)TEM Image of Pulverized Eggshell.



Figure 3: (a)XRD of Wood Charcoal. (b)XRD of Eggshell.

Table 1 presents the composition analysis of both the as-received grey cast and the carburized cast iron. Carbon and iron constitute a higher percentage of the elemental composition, which indicate the possibilities of obtaining a material of high strength and hardness after the heat treatment process (carburization). However, it was noted that chromium and nickel were present in the carburized sample, which may be as a result of their presence in the wood charcoal. Chromium presence would increase the hardness and strength of the substrate (grey cast iron) while nickel increases the impact strength as well reducing corrosive effects of other elements.

Sample	С	Si	Mn	S	Р	Cr	Ni	Fe
As-received	2.68	1.42	0.63	0.13	0.28	_	_	Bal.
Carburized	3.927	1.737	0.395	0.285	0.144	0.206	0.061	91.70

Table 1: Chemical Composition of Grey Cast Iron (As-Received and Carburized)

Figure 4 (a–c) show the scanning electron microscope (SEM) and x-ray dispersive (XRD) spectra of samples carburized at varying temperature of 700°C, 800°C and 900°C, respectively. Figure 4(a) show the SEM morphology and XRD spectrum of sample carburized at a temperature of 700°C. Porous structures were observed at the interface of the surface, which was due to the high carbon content and increased surface area. The pulverized eggshell has an accelerated effect on the diffusion of carbon into the substrate during the heat treatment process. Thus, increasing the bond strength of the carbon layers which will enhance its mechanical properties.

Additionally, graphite layers were observed at surface, which indicate the presence of carbons at the interface and this also increased the volume of mesoporous formation at the grain boundaries compared to other microstructures (Figure 1). The XRD pattern (Figure 4(a)) showed that there were broad peaks at 31.25°C and 61°C, respectively. It is clear that carbon particles diffused into the metal surface to improve the mechanical properties such as strength and hardness at that operating temperature.

The SEM and XRD results, for sample, heat treated at 800°C for three (3) hours which is presented on Figure 4(b). A more porous surface was observed irrespective of carburizing time. Hence, heat-treated surface was characterized by carbides, both at the inner and the outer surface. Also, observed is the thickness of the carburized surface at 800°C which was found to be more as compared to as-received substrate (Figure 1). This is due to the porous structure of the pulverized wood charcoal and eggshell which allows more absorption by the substrate (grey cast iron). XRD analysis of the carburized material at 800°C showed that broader peaks were obtained at a higher temperature. This is an indication of accelerated carbon diffusion at higher temperature.

Figure 4(c) presents the SEM morphology and XRD spectrum of carburized sample at 900°C. It was observed that at high temperature, carbon atoms diffused faster into the metal which resulted to graphite formation at the substrate interface. Thus, the entire surface is characterized by retained austenite, martensite and graphite layers. In addition, the interfaces of the microstructure (Figure 4(c)) showed that there was strong metallurgical bonding of the diffused carbon into the substrate. More so, the result of carburization at 900°C with a holding time of three hours show that there was large number of carbon deposit which promote graphite formation at the interface of the substrate metal.

More so, the micro-hardness values of the as-received substrate (grey cast iron) and the carburized samples at different temperature were systematically determined using Vickers hardness tester (Figure 5). The hardness values are a function of the concentration of carbon atoms.<sup>27</sup> Thus, the pattern of micro-hardness distribution is likened to the graded diffusion of carbon atoms. The micro-hardness of specimens carburized at 800°C and 900°C is greater than 260 HV, on the

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average. This suggests an enhanced hardening effect compared to as-received sample. Furthermore, the sample carburized at 900°C show higher micro-hardness than the other carburized samples and as-received, which indicates a better improvement in mechanical properties, thus better wear resistance.



Figure 4: (a–c) SEM Morphology and XRD of Grey Cast Iron Carburized at (a) 700°C (b) 800°C (c) 900°C for 3 h.



Figure 5: Micro-Hardness Values of the As-Received and Carburized Samples.

### **4. CONCLUSIONS**

Temperature and time were the major factors which influenced the diffusion process of carbon atoms into the substrate material (grey cast iron). The precipitation of the graphite on the substrate material induces a great improvement on the mechanical properties of the material characterized at varying temperature. Thus, carburizing temperature significantly influenced the diffusion of carbon atoms and in turn lead to formation of thick layers as well as hardness improvement, as it could be observed for the hardness values at 800°C and 900°C, respectively, compared to the value of as-received material. In summary, the microstructure and the mechanical characterization of the carburized samples demonstrate that pulverized wood charcoal and eggshell are good organic carbon for surface reinforcement of grey cast iron.

### **5. ACKNOWLEDGEMENT**

The authors are grateful to the management of Covenant University for the support of the research work.

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