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ORIGINAL RESEARCH ARTICLE

INFLUENCE OF ANTIMONY MICRO-ALLOY ADDITION ON THE MECHANICAL PROPERTIES OF CARBIDIC AUSTEMPERED DUCTILE IRON (CADI)

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ABSTRACT

In this research work, the influence of antimony micro-alloy addition on the hardness property, impact energy and wear resistance of carbidic austempered ductile iron (CADI) was investigated. Rod-shaped samples were produced using a sand casting technique to investigate the influence of antimony micro-quantities on the microstructural modification of the high manganese content (CADI). Selected mechanical properties of the product (CADI) have been tested. Rod-shaped samples were produced using a sand casting technique to investigate the influence of antimony micro-quantities on the microstructural modification of the high manganese content (CADI). Selected mechanical properties of the product (CADI) have been tested. Rod-like shape samples were produced by sand casting technique used for evaluating the influence of micro - quantities of antimony on the microstructural modification of high manganese content carbidic austempered ductile iron (CADI) with respect to the selected me 7chanical properties. Six compositions of carbidic ductile iron (CDI) with varying antimony content ranging from 0.096 % to 0.480 wt. % were produced, having equivalent carbon of hyper-eutectic composition (4.43). These samples were heated to austenitic temperature of 910°C and subjected to austempering temperature, 300°C for period of 1-3 hours. Microstructural examination of the samples was carried out to study the matrix and reinforcing phases present. The abrasion wear resistance was evaluated in accordance with the ASTM G 65 standard. The results show that; the obtained wear resistance values ranging from 2.35E8 to 3.29 E9 with respect to unmodified CADI (taken as reference material) The results show that the wear resistance values ranging from 2.35E8 and 3.29 E9 with respect to CADI sample without antimony addition taken as reference material, Rockwell hardness values obtained ranging from 28.6 – 55 HRc and impact toughness values ranges from 46.5 – 53 J for the developed CADI. Micrographs obtain revealed that chunky graphite, blocky carbides and pearlite phases were seen in the as-cast sample without antimony addition while the samples with varying addition of antimony element showed spiky graphite, granular carbide and more pearlite phases. However, after subjecting the samples to austempering processes, the pearlite phase transformed to ausferritic structure, while the carbide and graphite structure remained unaltered. The produced CADI is found to be useful for agricultural implements production.

1.0 Introduction

CADI is a material well known for its ability to survive different wear mechanisms such as rolling contact fatigue, adhesion and abrasion (Magalh˜aes and Seabra, 1998). It is well known of the ability of CADI to do well when it is subjected under different wear mechanisms such as rolling contact fatigue, adhesion and abrasion. Austempered ductile iron (ADI) act in diverse ways under abrasive conditions, based on tribo-system (low or high stress abrasion) involved in the process. Selection of proper heat treatment parameters couldtranslate to good performance of this material in service (Dommarco et *al.,* 2001). CADI, is a type of cast iron having carbides intentionally introduced in an ausferritic matrix. From metallurgical point of view, the presence of carbides in CADI is expected to promote an increase in the abrasion wear resistance, while compromising its impact toughness value. Available research revealed that the presence of carbides in CADI is expected to promote an increase in the abrasion wear resistance, while compromising its impact toughness value. In addition, there is the need to control the microstructure of CADI in order to obtain an optimum balance between abrasion resistance and impact toughness. One of the methods used in obtaining a microstructure of as-cast carbides in cast iron is to lower is to low the quantity of graphitizing elements (silicon) in the cast iron, to facilitate precipitation of ledeburitic carbides during its solidification. The above may be combined with a second option of putting a chill in the mould which promotes high undercooling. A third option is adding alloying elements (chromium, molybdenum or titanium) that are carbides stabilising in the melt (Hayrynen, and Brandenberg, 2003) which greatly narrows the interval between stable and metastable eutectic temperatures and promote total or partial solidification according to the metastable diagram Gundlach *et al.,* 1985). It must be noted that an under - cooling also affects the size and count of the solidification units, and thereby causing micro - segregation. The lower the cooling rate, the greater the micro - segregation effect, increasing the possibility of carbide precipitation at the last to freeze zones, and resulting to formation of alloyed carbides (Lacaze *et al.,* 2003). The size and composition of carbides can vary, from typical unalloyed ledeburitic to thin plate shaped high-alloyed carbides, based on the chemical composition and cooling rate of the material (Nastac and Stefanescu, 1997; Zhao and Liu, 2001). Caldera et al. (2005) reported that ledeburitic carbides produced either via controlling of cooling rate or the silicon level (non-alloyed carbides) have a high propensity to dissolve during the austenitizing stage and are not as stable as alloyed carbides.

Therefore, the objective of this work is to produce CADIs of varying micro-alloy addition of antimony (Sb) and identify its microstructural characteristics and evaluate some mechanical properties, such as their hardness values, abrasion resistance and the impact toughness.

2. Materials and MEthods

2.1 Experimental procedures

2.1.1 Preparation of Sample

The charged materials used in this work were sourced from a metal casting company. Indirect electric arc furnace of capacity 3 kg was used for melting of the charged materials. Locally sourced grey cast iron scraps were used as charge materials.

All the melts were nodulized with Fe-Si-Mg (9 wt. % Mg) and inoculated with FeSi (75 wt. % Si). Calculated amounts of Ferro-chrome, ferro-nickel, ferro-manganese, 99% purity copper, and antimony powder were added in the expected quantities after charge calculation was done. Six alloys of carbidic ductile iron were cast, with 0 wt. % Sb, 0.096 wt. % Sb, 0.192 wt. % Sb, 0.288 wt. % Sb, 0.384 wt. % Sb and 0.480 wt. % Sb content. The dimension of each sample cast is 20 mm by 20 mm by 200 mm. The rods were cut longitudinal into 20 mm by 30 mm long for the microstructural characterization, 10 mm thick by 50 mm long for impact and 20 mm x 40 mm for wear test samples. The six cast alloys were then heat-treated by heating them to austenitizing temperature of 910°C and held for 1 hour in a heat treatment furnace followed by an austempering step in an equal mixture of sodium nitrate and potassium nitrate salt bath at temperature 300 °C at austempering time of 1-3 hours.

2.2 Chemical and Microstructural Examination

The elemental compositions of the produced alloys were determined by means of a Spark Emission Optic Spectrometer (Spectro-pro MAXx-LMM06) produced in USA by Spectro Ametek with a DV6 excitation source. The microstructures of the produced alloys were accessed using light reflected optical metallurgical microscope (Axio-Observer A1m manufactured in USA by CIT NELIAN) complimented with FESEM Scanning Electron Microscope manufactured in China by i-CRIM with an EDX module, for the purpose of analyzing the influence of antimony element on the matrix and carbide formation of the produced samples. Metallographic preparation of the samples for microscopy examination was achieved using standard techniques for cutting, grinding and polishing, etching the polished samples with 2 wt.% Nital before viewing the prepared samples under microscope (Khanna, 2009)

2.3 Mechanical Tests

2.3.1 Wear Test

The abrasive wears resistance of the samples was measured using Rotopol-V tribometer manufactured by RTECH with Model number MFT-5000. The wear test which was done was based on the Taber method. Wear test that was carried out was based on Taber's method. This was done by attaching sample to a rotating horizontal disk of 25 cm radius and pressure of a specified load was applied according to Friedrich *et al.* (2009). The samples used were of dimensions, 15mm diameter by 40 mm long. The grit of emery paper used was 220, and revolution of 150 rpm for5 minutes. The weight of the samples in each case was measured before and after each test with a 4 decimal places Analytical electronic weighing scale with accuracy of 0.01 mg. After running through a fixed distance, the samples were removed, cleaned with acetone, dried, and weighed to determine the weight loss due to wear and the weight loss was converted to volume loss by using the material density measured, wear rate and wear resistance respectively.

Wear rate was measured using the equations

$$\text{Wear Rate} = \frac{Wv}{D} \quad (1)$$

Where;

Wv = Wear Volume (m^3)

D = Slide distance (m)

$$\text{where: Slide Distance } D = \frac{2\pi Rd}{60t} \quad (2)$$

and

R = Revolution = 150rpm

d = Diameter of the rotating disc used (m)

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Wear resistance is $\frac{1}{\text{wear rate}}$ (3)

Wear Resistance = inverse of wear rate obtained

Grit used is P 220 rough, revolution (R) is 150 rpm, d is the diameter of the rotating disc used.

2.3.2 Impact Test

Impact toughness tests was carried out on the produced CADI. The alloyed CADI and the heat-treated samples were carried out under the ASTM E 23 standard using Charpy method in which the samples were machined to square cross-section of 10 mm by 10 mm and 55 mm long, notched at the midpoint. The specimen was placed each in a vice as a beam fixed at two ends. The pendulum of 65 kg weight was raised, on releasing the hammer, the sample was hit behind its V-notch. The energy absorbed was read from the dial scale mounted on the machine.

2.3.3 Hardness Test

The hardness test for both as-cast and after heat treatment was conducted using Digital Rockwell hardness tester on scale C, in accordance with ASTM E18-08 (standard test method for Rockwell hardness of metallic materials). The minor load used for this test was 10 kg while the major load was 150 kg. The sample was placed on the anvil. The minor load was applied by moving the screw jack up until the specimen came in contact with the indenter. The hardness value was then displayed on the machine. Four points hardness values were measured and average of the four measurements was taken.

3. Results and Discussion

3.1 Chemical and Microstructural Characterization

Table 1 shows the chemical composition of the produced samples. It shows an approximately hyper - eutectic composition (carbon equivalent, C.E of 4.43).

There is no dissolution of carbide observed in the microstructure of the CADI after heat treating the CDIs (Caldera *et al*., 2005) this could be due to the high thermodynamic stability of alloyed carbides created by the high chromium content of the alloys. Figures 1 stands for as-cast microstructures of the CDIs which consist of chunky graphite nodules, blocky - carbide and pearlite phases while Figures 2, 3, 4, 5, 6, 8, 9, 10, 11 and 12 represent the austempered samples which compose of spiky graphite nodules, granular carbides and ausferritic matrix. From the micrographs obtained, the presence of antimony promoted pearlite formation of the alloy and causes improvement in the nodularity of the graphite nodule and increases hardness of the metallic matrix of the produced alloys (Patil et al., 2014) while the chromium assisted in the formation of alloyed carbide. After austempering, it was observed that the blocky and granular carbides were still retained in the structure, spiky graphite nodule was still present but no pearlite phase was observed, rather ausferritic matrix was observed. The carbide was stable during the austenitizing stage of the austempering, this is in conformity with (Patil *et al*., 2014).

Table 1: Chemical Composition of the Produced Samples

No	Sample	Fe	C	Si	Mn	Cr	Ni	Cu	Mg	Sb	S	P	C.E
1	C(Ctrl)	89.39	3.62	2.42	0.58	2.6	0.60	0.69	0.06	-	0.004	0.040	4.43
2	A	89.33	3.62	2.40	0.57	2.61	0.61	0.65	0.07	0.096	0.005	0.041	4.43
3	B	89.24	3.62	2.41	0.57	2.62	0.62	0.61	0.07	0.192	0.005	0.042	4.44
4	C	89.19	3.63	2.40	0.56	2.60	0.60	0.62	0.07	0.288	0.005	0.042	4.44
5	D	89.10	3.62	2.41	0.56	2.63	0.61	0.62	0.06	0.384	0.005	0.040	4.44
6	E	88.97	3.62	2.40	0.58	2.62	0.60	0.63	0.06	0.480	0.004	0.041	4.43

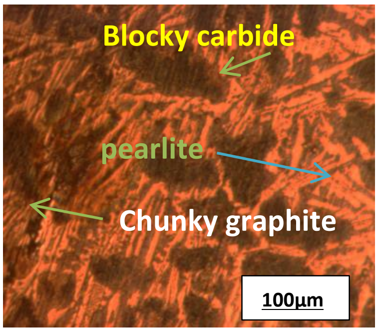

Figure 1: 0 % wt. Sb

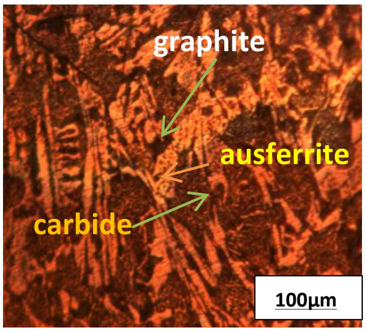

Figure 2: 0.12 %wt. Sb at 300 °C

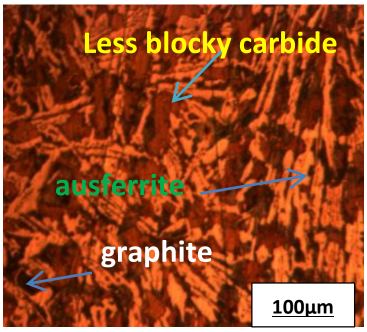

Figure 3: 0.24 % wt. Sb at 300 °C

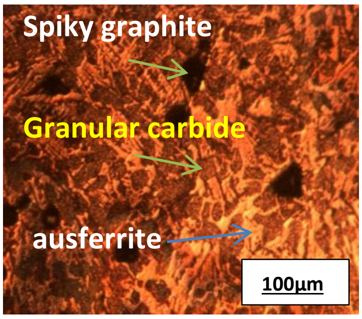

Figure 4: 0.34 % wt. Sb at 300 °C

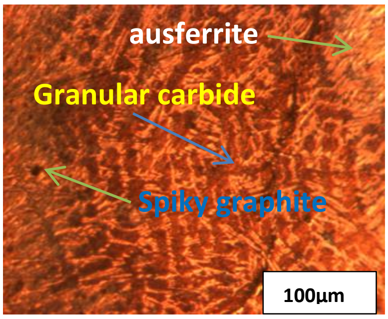

Figure 5: 0.41 % wt. Sb at 300 °C

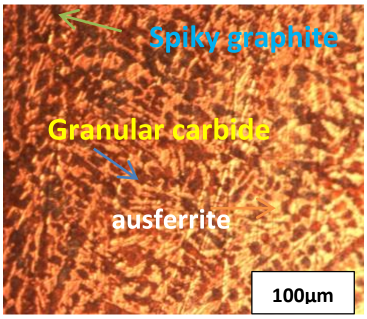

Figure 6: 0.48 % wt. Sb at 300 °C

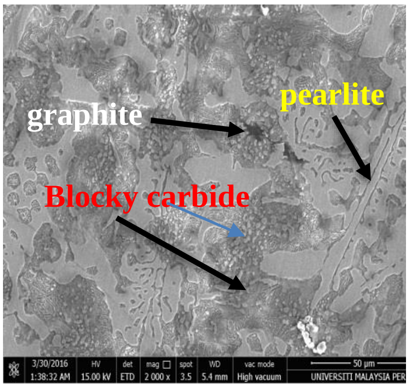

Figure 7: 0 % wt. Sb

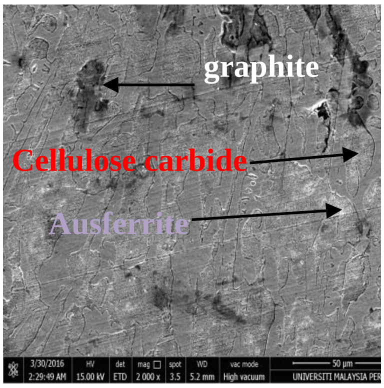

Figure 8: 0.12 % wt. Sb at 300 °C

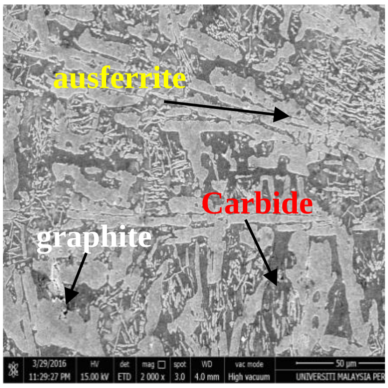

Figure 9: 0.24 % wt. Sb at 300 °C

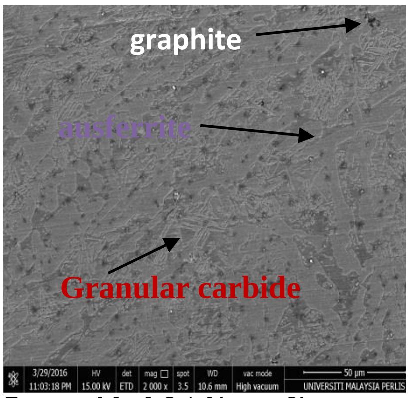

Figure 10: 0.34 % wt. Sb at 300 °C

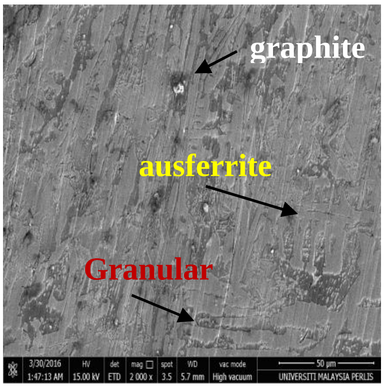

Figure 11: 0.41 % wt. Sb at 300 °C

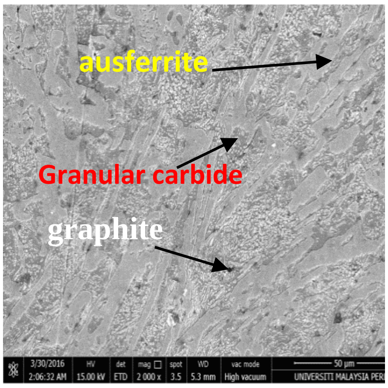

Figure 12: 0.48 % wt.Sb at 300 °C

The result shows no dissolution of carbide observed in the microstructure of the CADI after heat treating the CDIs (Caldera, 2005). This could be due to the high thermodynamic stability of alloyed carbides created by the high chromium content of the alloys. From the micrographs obtained, the presence of antimony promoted pearlite formation of the alloy and causes improvement in the nodularity of the graphite nodule and increases hardness of the metallic matrix of the produced alloys (Diao et al., 2011), while the chromium assisted in the formation of alloyed carbide. After austempering, it was observed that the blocky and granular carbides were still retained in the structure, spiky graphite nodule was still present but no pearlite phase was observed, rather ausferritic matrix was observed. The carbide was stable during the austenitizing stage of the austempering, this is in conformity with Patil et al. (2014).

3.2 Mechanical Test

3.2.1 Hardness Test

The Rockwell hardness value obtained for the control sample was 38HRc, while the highest hardness value of 52.5HRc was obtained gotten for the sample with 0.288 wt.% antimony, austempered at temperature of 300 °C for 3 hours. It was observed that there was an increase in the hardness values of the samples as the antimony content increased. This could have been caused by the promotion of pearlite phase in the alloys that was by the antimony element present in the produced CADI (Diao *et al.,* 2011).

A change in hardness values of the alloys with antimony content could be attributed to the variation in volume fraction of carbide and their sizes. Addition of more antimony into the CDI increases the volume fraction of the carbides which were smaller in size. Such variations can strengthen the material by impending dislocation movement more effectively. However, when the added antimony goes beyond certain amount (0.384 wt. % Sb), the hardness starts to decrease which is corresponding to the decrease in the volume fraction of carbides as observed in the present case. Finer carbides were found to be more effective to strengthen carbidic austempered ductile iron than coarse carbides.

A change in hardness values of the alloys (Figure 13) with antimony content could be attributed to the variation in volume fraction of carbide and their size. Addition of antimony element in the CDI increases the volume fraction of the carbides which were smaller in size. Such variations can strengthen the material by blocking dislocation movement more effectively. However, when the added antimony element goes beyond certain amount (0.384 wt. % Sb), the hardness starts to decrease, corresponding to the decrease in the volume fraction of carbides as observed in the present case. Finer carbides were found to be more effective to strengthen carbidic austempered ductile iron than coarse carbides.

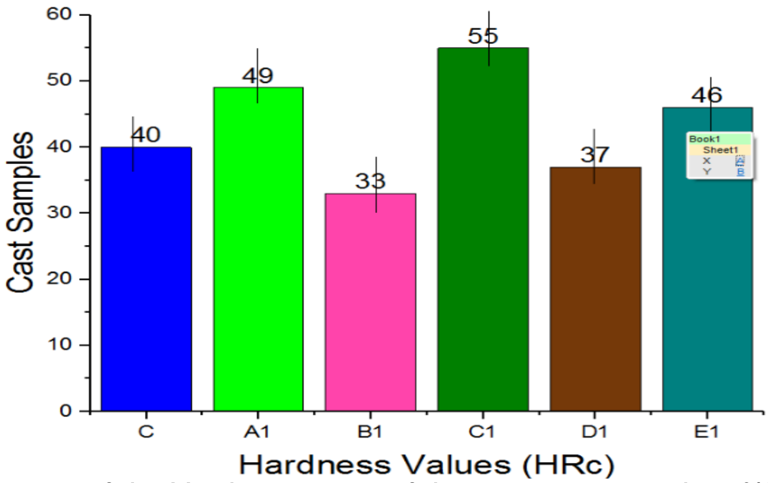

Figure 13: Variation of the Hardness values of the cast samples with wt%Sb for 1 hour austempering time for an Austempering Temperature of 300°C

3.2.2 Wear Test

The wear test was conducted in accordance with ASTM G-99 standard. The highest wear resistance value (Figures 14) was obtained in sample containing 0.34% Sb austempered at 300°C for 3hour which has wear resistance of 3.74E9 which is the optimum value for both wear and hardness properties of the samples. Weight loss was found to be as a result of the chromium content, critical content of antimony, carbon equivalent, austempering heat treatment parameters and microstructure matrix. The wear loss obtained was generally lower than the one obtained in previous research (Patil *et al.,* 2014). The wear resistance of the samples was found to increase with increase in antimony content, which is probably because of significant role which antimony element played by increasing the nucleation rate and inhibiting the deterioration of graphite nodules (Diao *et al.,* 2011). Hardness to large extent determines the abrasion resistance of carbidic austempered ductile and the changes in the wear rate were mainly affected by the carbide content. The hard carbides may have played main role in raising hardness and resisting abrasion under low contact stresses (Giacchi *et al.*, 2007). The abrasion wear resistance of this many-phases antimony modified CADI was influenced by hardness, shape, size, volume fraction and distribution of hard second phases (carbides) and properties of the matrix (Gahr, 1998). It is worth noting that the hardest sample did not show minimum wear rate, indicating that other factors (such as toughness) also have effect on the abrasion wear rate of this cast iron. The existence of fine granular carbides was seen to be of merit in terms of toughness than the ones of blocky carbide. The granular carbide suppresses micro-cracking more effectively due to its lower stress concentration. The added antimony promoted the formation of granular carbide but reduced the volume fraction of carbides. Ausferrite matrix in the produced material have also played a great role in the increasing the abrasion resistance of the CADI as obtained.

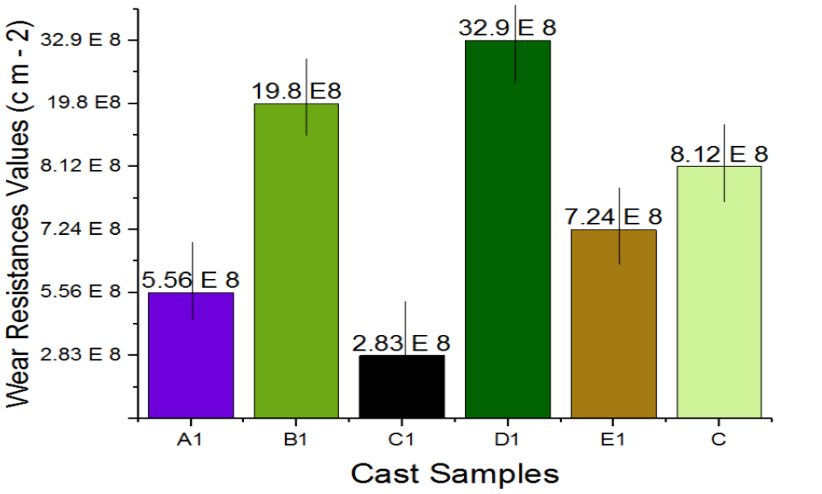

Figure 14a: Variation of the Wear Resistance Values of the cast samples with wt%Sb for 1 hour austempering time for an Austempering Temperature of 300^{0}C

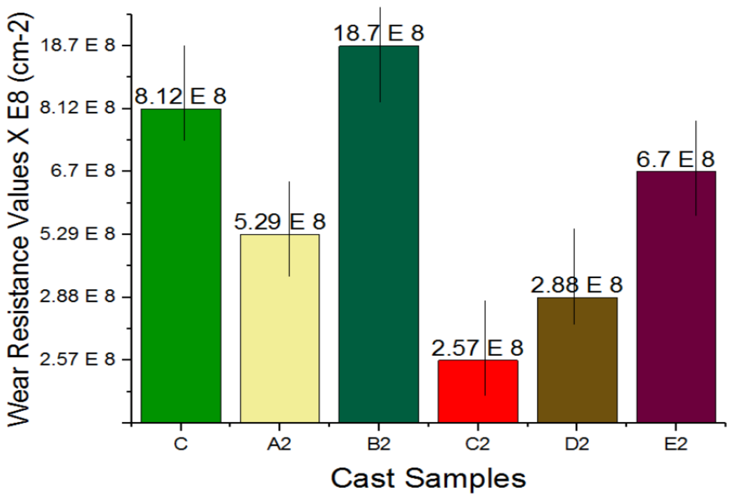

Figure 14b: Variation of the Wear Resistance Values of the cast samples with wt%Sb for 2 hours austempering time for an Austempering Temperature of 300^{0}C

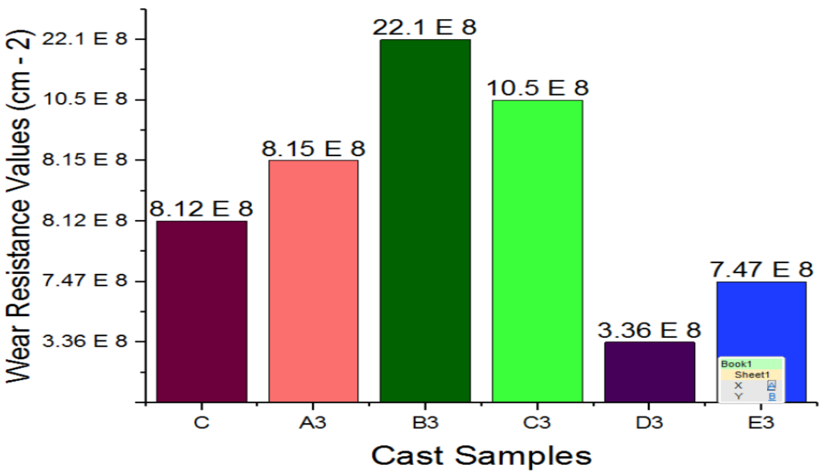

Figure 14c: Variation of the Wear Resistance Values of the cast samples with wt%Sb for 3 hours austempering time for an Austempering Temperature of 300^{0}C

3.2.3. Impact test

The results of the impact toughness (J) versus antimony content in all the material variants studied are shown in Figure 15.

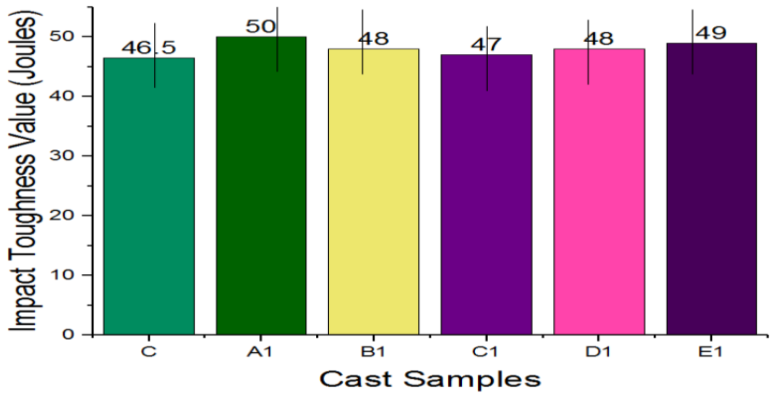

Figure 15: Variation of the Impact Toughness Values of the cast samples with wt%Sb for 1hour austempering time for an Austempering Temperature of 300°C

From the impact toughness results obtained, there is marginal increase in the impact energy as the antimony content and austempering time increase from 1 to 3hours. This indicates that increase in antimony content led to increase in impact toughness as a result of modification of the matrix phase of the produced carbidic ductile iron and the refinement of the graphite nodules (Diao *et al.*, 2011). Increase in austempering time also translates to increase in impact energy. This could be as result of the increase in the ausferrite in the matrix of the samples. Granular carbide as earlier said is of advantage in terms of toughness and resistance to fatigue crack propagation when compared to coarse carbide. The eutectic carbide crystallizes at the γ-liquid phase boundary. The added antimony element may have preferentially stayed at the interface between γ – phase and carbides and hence discouraging the continuous growth of eutectic carbide. It also decreases the surface tension of the melt, thereby, encouraging the granular growth and stabilizing the granular interface (Sun *et al.*, 2012).

4. Conclusion

The produced Carbides were stable alloyed carbides. Antimony element added was found to have assisted in changing the graphite nodule from chunky to spiky structure. This helped in getting better mechanical properties.

The presence of granular carbides in the microstructure as a result of micro amount of antimony helped in increasing the hardness, wear resistance, and impact toughness of the produced alloy. Increase in the antimony content led to more granular carbides, the impact toughness of the alloys was improved. The highest wear resistance was obtained at 0.288 wt. % antimony content. The hardness results were not consistent with the wear resistance results. The best sample obtained is the carbidic austempered ductile iron with antimony critical content of 0.288 wt. %.

The analysis of the hardness, wear tests and the impact toughness results showed that the alloy with 0.384% Sb (0.384 - 300 -1hr) have a good balance between hardness, wear resistance and impact toughness, under the current experimental conditions.

These material variants have shown an important improvement in abrasion resistance with respect to the CADI variants, while at the same time show good impact toughness and hardness. This confirms that the produced CADI is found to be useful for agricultural implements production.

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