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EFFECT OF ANTIMONY ON THE WEAR RESISTANCE AND IMPACT TOUGHNESS OF CARBIDIC AUSTEMPERED DUCTILE IRON (CADI)

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Abstract

This work investigated the effect of antimony on the impact toughness and wear resistance of carbidic austempered ductile iron (CADI). Six alloys with a carbon equivalent value of 4.44 (hyper-eutectic composition) were used to evaluate the produced CADI, with antimony content ranging from 0.096 to 0.48 wt. %. The produced carbidic ductile irons (CDI) were heated to austenitic temperature of 910^oC and held for 1 hour, then austempered at temperature of 325^oC and held at different times (1-3 hours) to obtain carbidic austempered irons. Microstructural characterization was then carried out. The wear resistance was evaluated using a wear tester, it was noted that the wear resistance values of the CADI ranged from 2.35E8 cm^{-2} to 3.74E9 cm^{-2}. The impact toughness of the CADI was determined using Charpy method, the results obtained ranged from 46.5 – 55J. It was observed that the as-cast CDI has graphite nodules, blocky carbides with a pearlitic matrix, the samples with antimony addition had granular carbides, the heat-treated samples had their matrix transformed to ausferrite while other structures remained unchanged. Sample with antimony content of 0.288 wt. % has the highest wear resistance value while sample with 0.096 wt. % has the best impact toughness value.

Keywords: *Carbidic austempered ductile iron; Microstructure; Antimony; Wear; Impact toughness; SEM.*

Introduction

Austempered Ductile Iron (ADI) is popular for its high tensile strength (1600MPa) and impact toughness (100J). This property gives the material the capacity to compete favourably with forged steel in many areas of application. This material has been used where various wear mechanisms are needed [1–3]. ADI can serve well in many areas of usage if the proper selection of heat treatment parameters is carried out [2]. However, the wear resistance of ADI under high load is said to be non-ideal for applications like agricultural machinery or mining etc [4, 5], in a bid to upgrade this property (wear), some researches have led to the birth of a new type of ADI, containing carbides immersed in an ausferritic matrix, called carbidic ADI or CADI. It has better wear resistance than ADI.

The role of alloying elements and cooling rate on some mechanical properties and microstructures of CADI have been published in the past years [6], fine bainitic plates with outstanding all-encompassing mechanical properties have been proved [7, 8]. Formation of bainites normally involve long holding time at low temperature. Multi-pass rolling before bainitic transformation [9], this substantially decreases production efficiency. The possibility of adopting

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super-high austenitizing temperature (960-100 ºC) to improve the impact toughness of CADI has been tried by researchers [10].

It was observed that super-high temperature pre-treatment can greatly decrease co-rich eutectic carbides at the grain boundary, and encourage the precipitate of particles in the prior austenite grains during reheating, much reduction of lamellar space of pearlite by super-high temperature, increase in reheating temperature (720 - 880 ºC) the pearlite reduces gradually, and the precipitated particles are retained, 12% improvement in impact toughness was achieve by super-high temperature [11, 12].

Austempering process on ductile cast iron do induce dominance of ausferrite, this ausferrite is a combination of high-carbon austenite (γ_{HC}) and acicular ferrite with graphite nodules in metallic matrix [1]. Austempering heat treatment can improve the impact energy, yield and tensile strength, and hardness of ADI [2].

Chromium addition to ADI reduces the stability of austenite, promote martensite transformation during cooling, improve the hardness and reducing elongation [13]. Studies have been carried-out on various materials or heat treatments to improve the hardness of lobes in camshafts by using steel with surface hardening of cams of the engine camshaft [14]. The microstructural features and mechanical properties of ductile iron low alloyed with 0.2 and 0.3 wt% of vanadium was studied on camshaft. The highest carbide formation (< 1wt.%) was mainly heated in the middle region of the lobes due to the inverse chill behaviour [14].

The presence of carbide in CADI promotes an increase in abrasion resistance, but on the other hand, toughness is compromised. Therefore, the critical issue relating to the development of this material is how to control its microstructure to obtain an optimum balance between abrasion resistance and toughness. One of the production routes of carbidic ductile iron is to alloy the grey cast iron melt with carbide stabilising elements like chromium, molybdenum, or titanium [15, 16].

This significantly reduces the gap between stable and metastable eutectic temperatures, which aids in forming the total/partial solidification of ferrous materials [17]. The composition and size of carbides can metamorphize from unalloyed ledeburite to thin plate-shaped high-alloyed carbides based on the chemical composition and cooling rate [17–20]. It has been observed that ledeburitic carbides can be created by controlling the cooling rate of cast iron or the silicon level (non-alloyed carbides). However, this unalloyed carbide has a high propensity to dissolve during the austenitization stage of the austempering process and is not as stable as alloyed carbides [21]. The purpose of this work is to produce different variants of micro-alloy of antimony (Sb) on CADI, study its microstructural characteristics, and evaluate some of its mechanical properties, particularly the abrasion resistance and impact toughness. This work is targeted at producing a material that is suitable for the production of agricultural implements and mining equipment at a lower cost and with an improved life span.

Materials and Methods

The materials used in this study were produced in a tertiary institution-based metal casting laboratory housing a 3 kg capacity indirect electric arc furnace, which was used to achieve the melt. Cast iron scrap of known chemical composition (graphite, ferrochrome and ferronickel) were used as charge materials after charge calculations were carried out following standard procedure as in Equation 1 [22].

$$\begin{pmatrix} Amount\ of\ Alloying \\ Element\ to\ be\ added \end{pmatrix} = \frac{\begin{pmatrix} Required \\ Amount \end{pmatrix} - \begin{pmatrix} Amount\ in\ the \\ base\ metal \end{pmatrix} \times \begin{pmatrix} Total\ wt.of \\ the\ charge \end{pmatrix}}{Purity\ of\ the\ Alloying\ Element} \tag{1}$$

In all the castings, the melts were nodulized with Fe-Si-Mg (9 wt. %Mg) and inoculated with FeSi (75 wt. % Si), calculated micro-quantities of antimony were later added in the ladle and the melt was thoroughly mixed before pouring in a prepared sand mould. Six alloys of the carbidic ductile iron were produced with varying antimony content of 0 wt.% Sb, 0.096 wt.% Sb, 0.192 wt.% Sb, 0.288 wt.% Sb, 0.384wt.% Sb and 0.48 wt.% Sb. The dimension of each cast sample was 200 mm in length, 20 mm in breadth and 20mm thick. From all the cast samples produced, respective sectional dimensions were cut into 20 mm by 30 mm; 10mm thick by 50 mm; 20 mm by 40 mm and 20 mm diameter by 25 mm length to carry out microstructural characterization, impact toughness test, wear test and hardness measurement respectively.

Five of the produced antimony-modified carbidic ductile irons were then subjected to austempering heat treatment process by heating the samples to austenitization temperature of 910ºC and held for 1 hour in a muffle furnace for homogenization, followed by an austempering step which was carried out in a sodium nitrate - potassium nitrate salt bath at a temperature of 325ºC for between one to three hours.

Chemical and microstructural examination

The chemical composition of the produced alloys was determined with the aid of a spark emission optic spectrometer with a DV6 excitation source. The microstructure of the cast samples was evaluated using a FESEM Scanning Electron Microscope with an EDX module, to analyse the effects of the antimony and its influence on the carbide size, and phases after heat treatment operations. Metallographic sample preparation for optical microscopy examination was carried out on the samples using standard techniques for cutting, grinding and polishing before etching them with 2% Nital [23].

Wear test

The wear resistance of the samples was evaluated using the Rotopol-V Wear measuring machine according to the ASTM G 65 standard. The surfaces of the samples were made parallel to the surface of the rotating disc of the machine. Emery paper used was Grit 60, and the revolution was set at 120 RPM. Care was taken to ensure that the samples under test were firmly held to the surface of the rotating disc during the test so that the samples do not fling out while running the test. The weight loss values obtained from the test using an analytical weighing balance of 4-decimal places with a precision of 0.1 mg were then converted into volume loss using the material density measured [24]. Wear rate (Wrate), wear resistance (Wres), sliding distance (S), Wear volume (V), radius (r) and time (t) were measured using expressions in Equations 2, 3 and 4.

$$Wear\ rate = \frac{\mathrm{V}}{\mathrm{S}} \quad (2)$$

$$\text{Sliding distance} = \frac{2\Pi \mathrm{r}}{60\times \mathrm{t}} \quad (3)$$

$$\text{Wear resistance} = \frac{1}{\mathrm{V}} \quad (4)$$

Impact test

Impact test was carried out to determine the impact toughness of the samples. Charpy method was adopted in which samples (test piece) of a square cross-section of 10 mm by 10 mm and 50mm long, notched at the midpoint were used. The specimen was placed in a vice as a beam fixed at the two ends. The pendulum of 65 kg weight was raised, and on releasing it; the sample was hit from behind the V – notch. The energy absorbed was read from the dial scale mounted on the machine according to Mahran [25]. Impact toughness test on the carbidic ductile iron, antimony modified carbidic ductile iron and the heat-treated samples were carried out under the ASTM E 23 standard.

Results and Discussion

Chemical and microstructural characterizations

The chemical compositions of the produced antimony modified CADI is shown in Table 1.

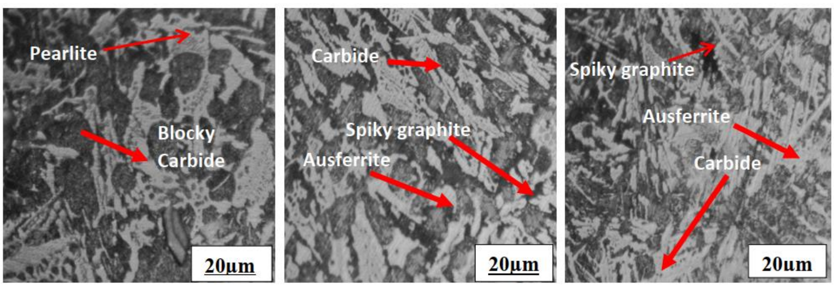
Table 1. Chemical composition of the produced CADI evaluated (wt%)

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

Carbon equivalent (C.E) calculated from the chemical composition of the produced alloys is 4.44 (hyper - eutectic).

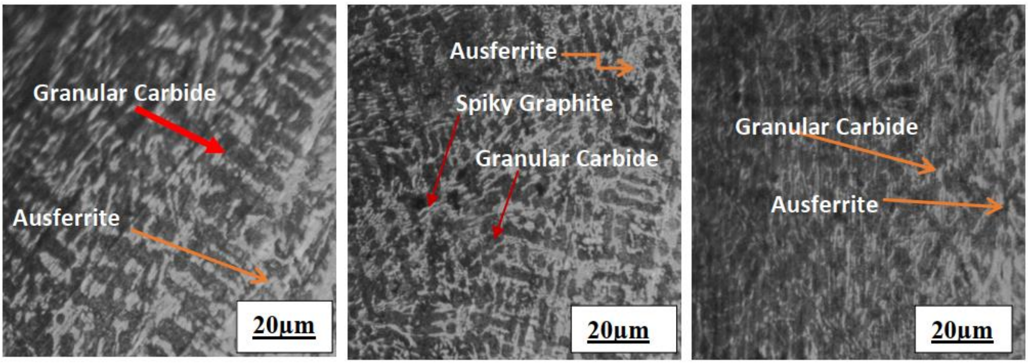
$$C.E = \%C + \frac{\%Si + \%P}{3} \tag{5}$$

From the microstructure obtained for the produced antimony modified CADI, there is no evidence of carbide dissolution after the austempering heat treatment operation which is in line with what was observed in literature [10], this indissolubility of the alloyed carbide in the CADI shows its high thermodynamic stability as a result of its high chromium content. Figures 1 and 7 represent as-cast microstructures which consist of form IV graphite nodules, blocky- carbides and pearlite matrix phase for the sample without antimony addition. While the samples with antimony addition possess form IV to form VI graphite nodules, granular and spiky carbides and pearlite matrix phase. The nature of the graphite formed in the alloys was a result of excessive antimony addition [26]. The Pearlite phase was found to be promoted in the microstructure of the samples as the antimony content increased [27]. Figures 2 - 12 represent the austempered samples which are composed of graphite nodules and granular carbides with an ausferritic matrix. This shows that antimony assists in pearlite formation of the carbidic ductile iron and improves the nodularity of the graphite nodules and increases the hardness of the phase [28]. After the austempering processes, it was observed that the blocky carbide and granular carbide were still retained in the structure, so also the graphite nodules were present but no pearlite phase was observed, rather, an ausferritic matrix was formed.



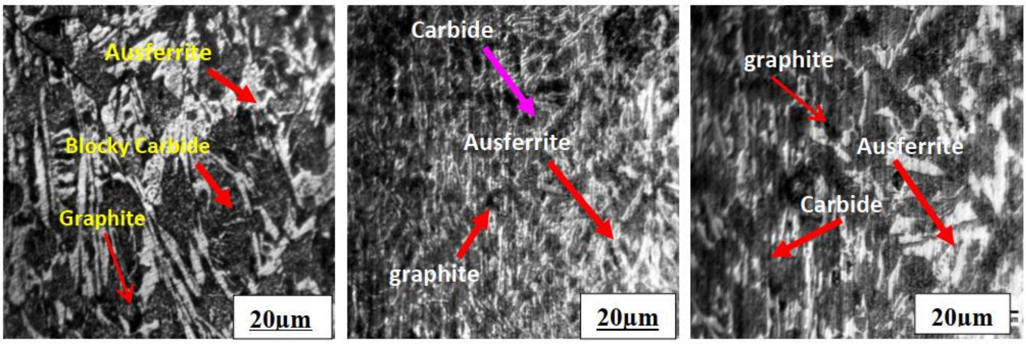
a) b) c)

Fig. 1. CDI/CADI with different content of Sb: a) 0.00% wt. Sb; b) 0.096 % wt. Sb austempered at 325ºC; c) 0.192 % wt. Sb austempered at 325ºC



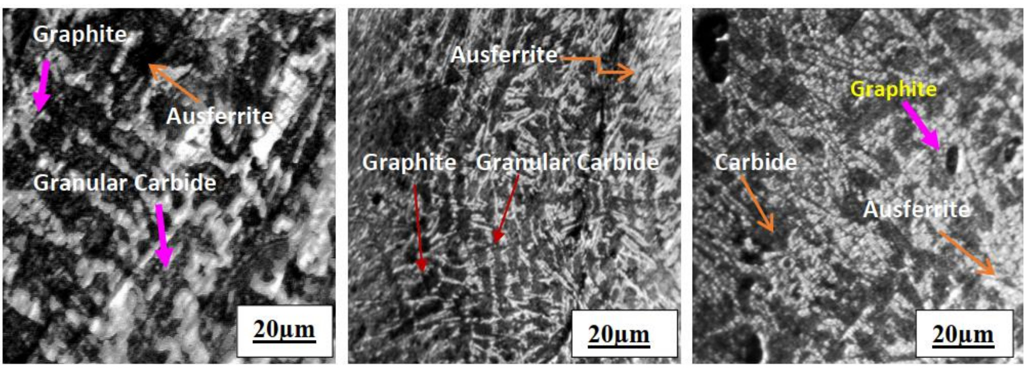
d) e) f)

Fig. 1. CADI with different content of Sb: d) 0.288% wt. Sb Austempered at 325ºC; e) 0.384% wt. Sb Austempered at 325ºC; f) 0.480% wt. Sb Austempered at 325ºC



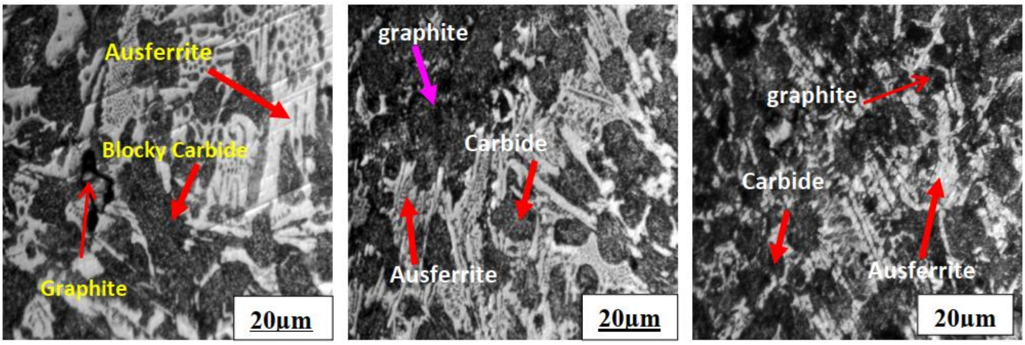
a) b) c)

Fig. 2. CDI/CADI with different content of Sb: a) 0.00% wt. Sb; b) 0.096 % wt. Sb austempered at 325ºC; c) 0.192 % wt. Sb austempered at 325ºC



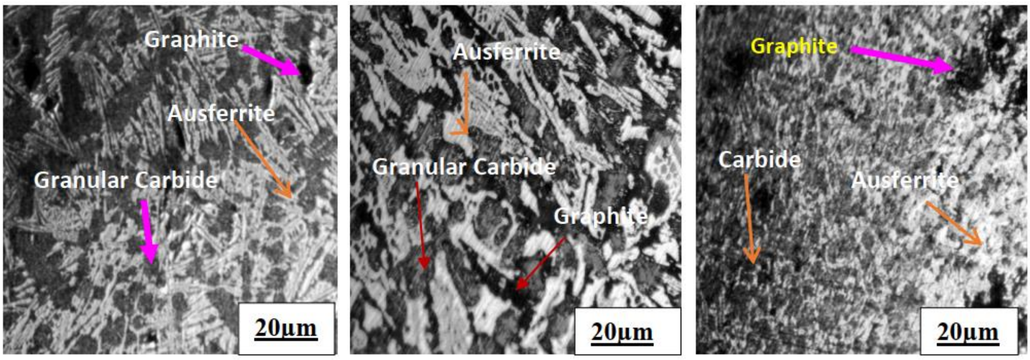
d) e) f)

Fig. 2. CADI with different content of Sb: d) 0.288% wt. Sb Austempered at 325ºC; e) 0.384% wt. Sb Austempered at 325ºC; f) 0.480% wt. Sb Austempered at 325ºC



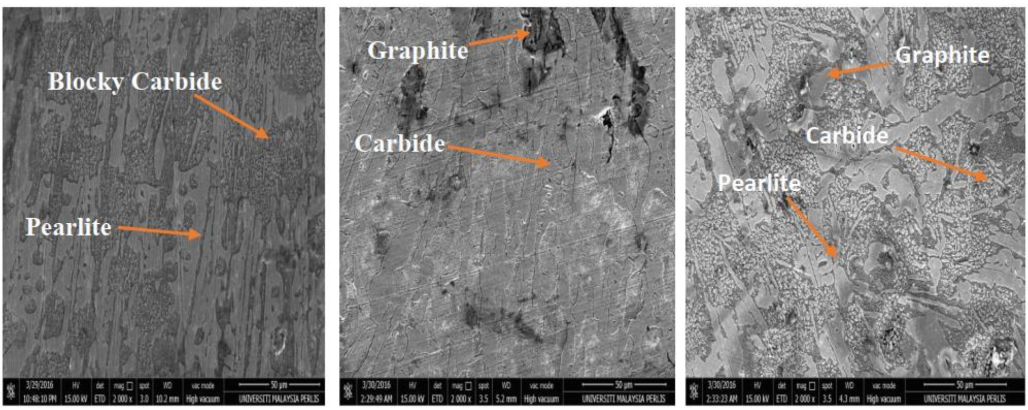
a) b) c)

Fig. 3. CDI/CADI with different content of Sb: a) 0.00% wt. Sb;
b) 0.096 % wt. Sb austempered at 325ºC; c) 0.192 % wt. Sb austempered at 325ºC



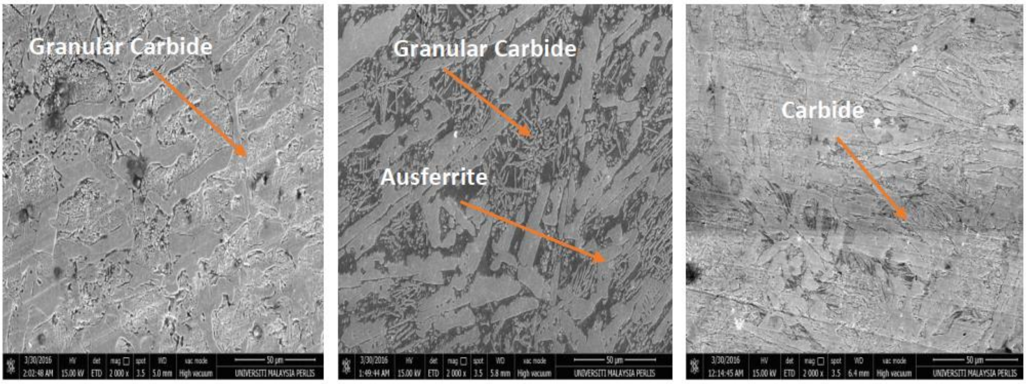
d) e) f)

Fig. 3. CADI with different content of Sb: d) 0.288% wt. Sb Austempered at 325ºC;
e) 0.384% wt. Sb Austempered at 325ºC; f) 0.480% wt. Sb Austempered at 325ºC



a) b) c)

Fig. 4. SEM of CDI/CADI with different content of Sb: a) 0.00% wt. Sb;
b) 0.096 % wt. Sb austempered at 325ºC; c) 0.192 % wt. Sb austempered at 325ºC



d) e) f)

Fig. 4. SEM of CADI with different content of Sb: d) 0.288% wt. Sb Austempered at 325ºC; e) 0.384% wt. Sb Austempered at 325ºC; f) 0.480% wt. Sb Austempered at 325ºC

Impact Toughness

The results of the impact toughness values obtained for all the produced samples studied are shown in Figures 5-8.

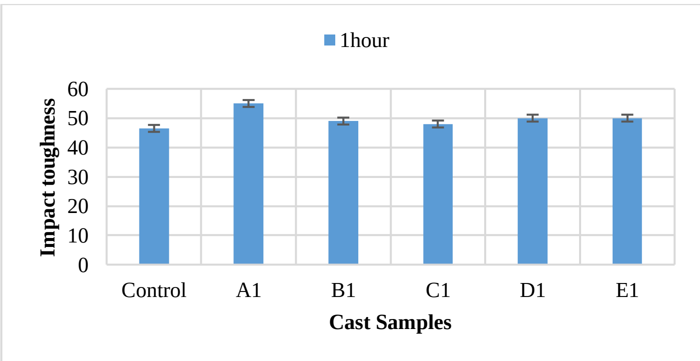


Fig 5. Impact toughness of the produced CADI samples with austempering temperature and time of 325ºC and 1 hour respectively.

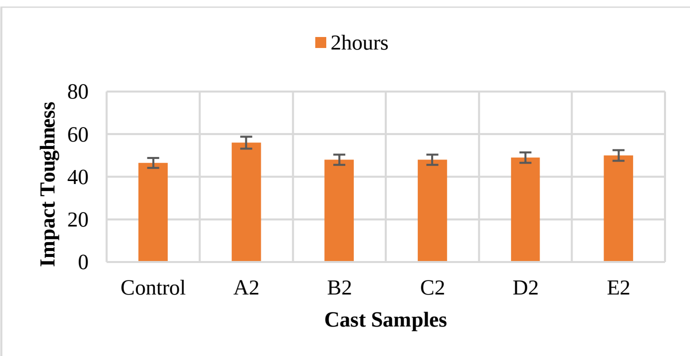


Fig 6. Impact Toughness of the produced CADI samples with austempering temperature and time of 325ºC and 2 hours respectively.

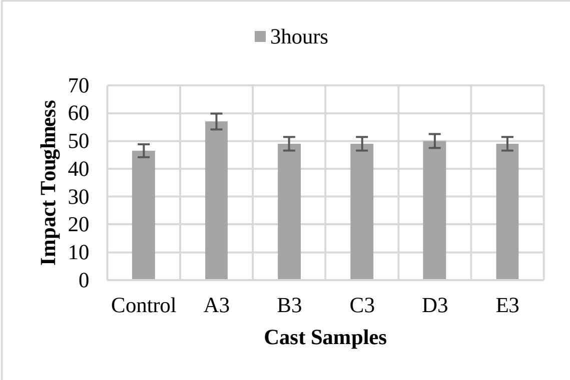


Fig 7. Impact toughness of the produced CADI samples with austempering temperature and time of 325ºC and 3 hours respectively.

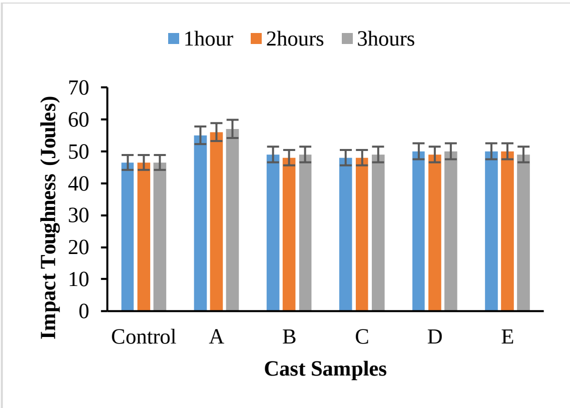


Fig 8. Impact toughness of the produced CADI samples with austempering temperature and time of 325ºC and 1-3 hours respectively.

From the figures, it was observed that sample A possesses the highest value of impact toughness with 55J compared with the control sample which has the lowest value of 46.5J. Hence, there was no appreciable decrease in the impact energy values as antimony content increased from 0.096 wt.% Sb to 0.48 wt.% Sb with austempering temperature and time. This indicates that the increase in antimony content in all samples does not have a negative effect on the impact toughness of the produced CADI, as a result of modification of the graphite shape in the phase. The granular carbides seen in the microstructure of antimony-modified carbidic austempered ductile iron was beneficial to impact toughness and resistance to fatigue crack propagation, compared to the unmodified CADI which has coarse carbide. The added antimony may have preferentially stayed at the interface between the γ – phase and carbides and hence decrease the continuous growth of eutectic carbide. It could have decreased the surface tension of the melt, so favouring the granular growth and stabilising the granular interface by preventing the growth of any micro-perturbation on the surface [16]. So also, granular carbides help minimise the stress concentration, nucleation and propagation of cracks and hence enhance the toughness. The refining effect of Sb on graphite nodules and carbide has greatly contributed to the improvement in the toughness [17]. Generally, as the antimony addition increases, the impact toughness decreases.

Wear property

The results of the wear resistance values obtained for all the produced samples studied are presented in Figures 9 - 12. The highest wear resistance value of 37.4 × 10^8 cm^{-2} was obtained for CADI sample C containing 0.288 % wt. antimony addition that was austempered at austempering temperature and time of 325ºC and 1 hour respectively; while the unmodified CADI sample (without antimony) has a wear resistance value of 8.12 × 10^8 cm^{-2} as shown in Figure 9. Meanwhile, CADI sample E possesses the least values at 325ºC and at all austempering times (i.e., 1 – 3 hours) as presented in Figure 12.

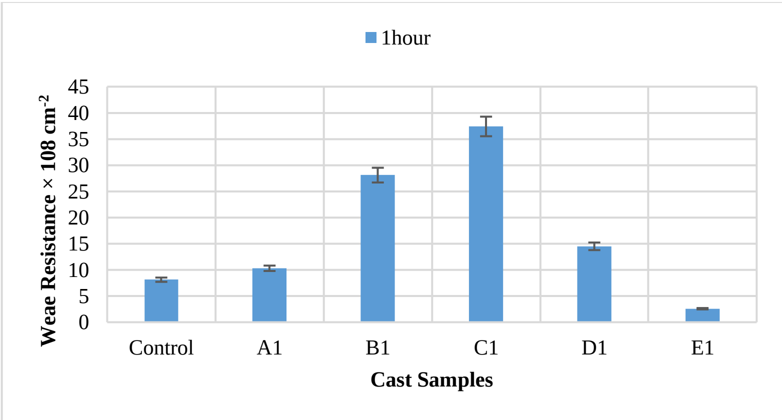


Fig 9. variation of wear resistance of the produced CADI samples with austempering temperature and time of 325 ºC and 1 hour respectively.

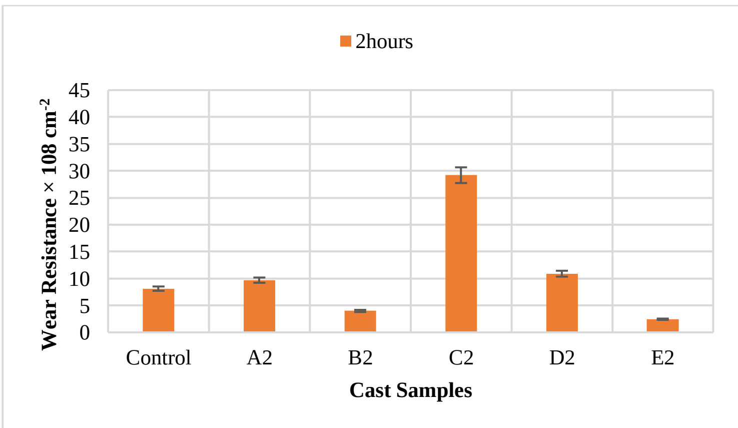


Fig 10. Variation of wear resistance of the produced cadi samples with austempering temperature and time of 325ºC and 2 hours respectively.

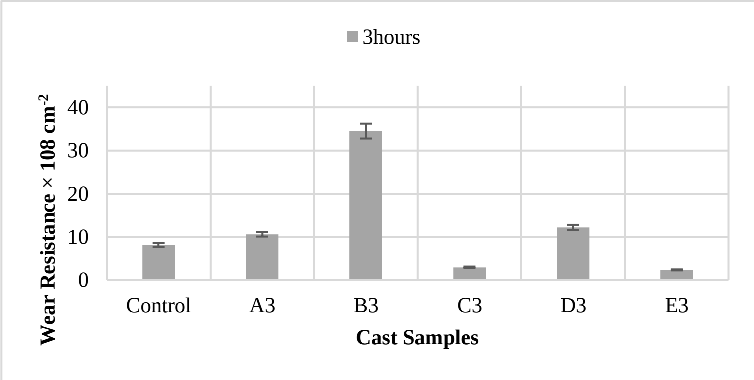


Fig 11. Variation of wear resistance of the produced cadi samples with austempering temperature and time of 325ºC and 3 hours respectively.

The abrasion wear resistance of this multiphase material (antimony modified CADI) was influenced by the shape, size, volume fraction and distribution of hard second phases (carbides) and the matrix [29]. It was observed that wear resistance of the produced antimony modified CADI was influenced by the ausferrite in its matrix and the carbide type. The existence of fine granular carbide [30] was helpful in blocky dislocation movement than blocky carbide ones. Also, the granular carbide caused by the micro-alloying of antimony in the produced CADI could have suppressed micro-cracking more effectively due to its correspondingly lower stress concentration. Thereby causing improvement in the wear resistance of the antimony modified CADIs.

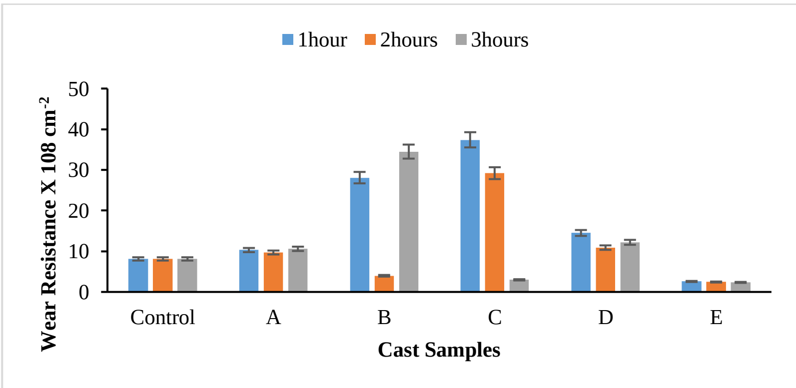


Fig 12. Variation of wear resistance of the produced CADI samples with austempering temperature and time of 325ºC and 1 - 3 hours respectively.

Conclusions

The following conclusions were drawn from the studies:

- The antimony contents range between 0.096 wt. % and 0.48 wt. % was produced by sand casting technique. The carbide was stable during the austenitizing stage of the austempering heat treatment process and the amount of dissolved carbides was negligible.

- The presence of carbides in the microstructure together with micro - amount (max. of 0.480 wt. %) of antimony increases the wear resistance without corresponding decreases in impact toughness.
- Increase in the antimony content led to granular carbides, spiky-graphite nodules and ausferritic matrix at austempering temperature of 325ºC.
- After the austempering process, the pearlite phase was observed to have transformed to ausferrite, while other phases present in samples remained unchanged.

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