The Effects of 0.16 % Chromium + 1.32 % Nickel Addition on Austempered Ductile Iron for Mechanical Properties and Slurry Erosion

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ABSTRACT

The effects of mechanical properties and slurry erosive wear behaviour on combination of Chromium and Nickel Alloyed Ductile Iron before and after austempering process were investigated in this study. Specimens of pure ductile iron and addition of 0.16 % Cr+ 1.3 2% Ni alloyed ductile iron were produced through conventional CO_2 *sand casting method. The specimens were then austenitized at 900* ^0C *before being tempered at 300* ^0C *for 40 minutes. The slurry erosion of newly developed material were obtained by means of slurry erosive wear and the mechanical testing involved tensile test (TS 138 EN1002-1), Vickers hardness test and Charpy Impact test (ASTM E23). Roughness tests as well as SEM observations were also done to Cr-Ni alloyed ductile iron samples. All the testing was done to both as cast and austempered specimens. Austempering process heat treatment of Cr-Ni alloyed ductile iron has shown an increase in slurry wear and mechanical properties compared to as cast due to solid strengthening effect of Chromium and Nickel. Meanwhile, the austempering process does improve the hardness of alloyed ductile iron.*

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Introduction

Ductile iron is also known as nodular cast iron or spheroidal graphite cast iron because of the presence of nodules/spheroids of graphite instead of graphite flakes which present in ordinary gray cast iron [1]. Ductile iron is an iron with the composition of gray iron in which the molten metal is chemically treated before pouring to cause the formation of graphite spheroids rather than flakes [2]. Alloying elements can strike constitution, characteristics and behaviour of the steel [3]. They interact with iron, carbon and other elements in the steel, resulting in alterations in mechanical, chemical, and physical properties of the steel. The point to which the properties of steel are changed by alloying depends on the amount of alloying elements introduced and the role of their interaction with the primary elements of the steel, i.e. Fe and C [4].

Austempered ductile iron happens when ductile iron is austempered in the temperature range of 230 to 370 °C (450 to 700 °F) and in the range of half an hour to four hours depending upon the composition and the desired physical properties [5]. Austempered ductile iron (ADI) provides many advantages compared to the quenched and tempered ductile iron. It demonstrates excellent combination of strength, toughness and ductility. It is expected to be attractive in equipment for other industries such as mining, earthmoving, agriculture and machine tools, by the same improved performance and cost savings [6]. ADI has higher strength and better ductility than standard ductile irons. This allows the part to be smaller and lighter and makes ADI desirable for uses such as automotive gears, crankshafts, and structural members for construction and transportation equipment, replacing wrought or cast steels [7].

In present, there was little evidence on the effect of both Chromium and Nickel addition on the properties of ductile iron and austempered ductile iron. This research however, penetrated on the effect of 0.16 % Chromium and 1.32 % Nickel addition on the mechanical and slurry erosion wear characteristics of alloyed ADI.

Experimental Procedure

Sample Preparation

Samples were casted via CO_2 sand casting method, in 60 kg capacity induction furnace until it reached the melting temperature of 1450 ºC. Nodularization process using Fe Si Mg was done before pouring process. The samples were produced in double-cylinder shape with dimension of 300mm long and 25±2 mm diameter both. The chemical composition range of ductile iron (DI) and 0.16% Chromium + 1.32 % Nickel alloyed ductile iron (analyzed using Ametek Spectromaxx Analytical tester) given in **Table 1**.

Table 1: Chemical composition of ductile iron sample (wt%)

Element	C	Si	Mn	P	S	Ni	Cr	Cu	Mg	Fe
Un-alloyed DI	3.46	1.76	0.42	0.07	0.01	-	0.001	0.02	<0.001	Bal
0.16Cr+1.32Ni Alloyed DI	3.40	1.62	0.41	0.04	0.01	1.32	0.160	0.45	<0.001	Bal

Heat Treatment

The austempering heat treatment process has been done by austenized the alloyed ductile iron to 900 ˚C for one hour using Carbolite furnace machine before quenching it inside a salt bath liquid at 350 ºC for 40 minutes. Specimens were subsequently withdrawn and cooled by nature air to room temperature. The schematic diagram of austempering process is shown in Figure 1.

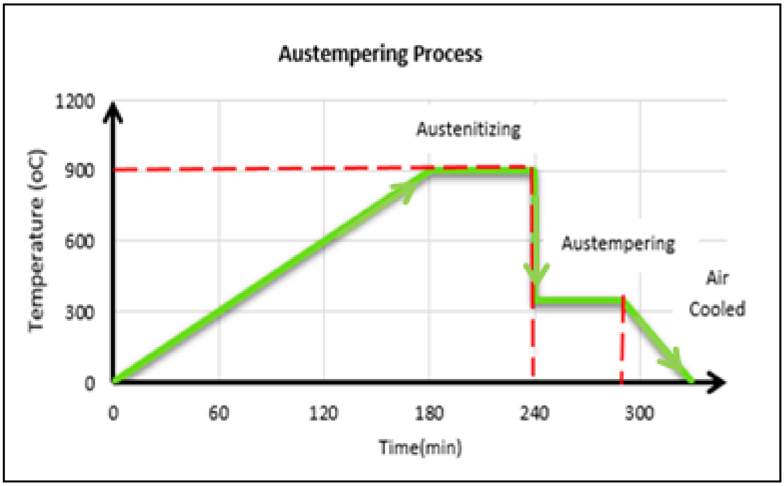

Figure 1. Schematic diagram of austempering process

Mechanical and Physical Testing

The mechanical testing involved tensile test (TS 138 EN1002-1), Vickers hardness test and Charpy Impact test (ASTM E23). At least three specimens were tested representing each composition and austempering holding times and only average values were reported.

Slurry Erosion Wear and Roughness Test

The erosive wear specimens with dimension of 76 mm (length) x 25 mm (width) x 6 mm (height) were first ground on belt emery to ensure the surface was flat on either side. A 6 mm hole was drilled in each sample purposely to mount the samples in the equipment. This test was run by using the slurry erosion test machine which consisted of stirrers and can held six samples at a time with the help of shackles. The slurry mixture contained of silica sand of nominal size 1.18 mm and water with ratio of 30:70. The pH level of water was kept at 7.5 by adding sodium chloride. All the samples were dipped in slurry water and silica sand and stirred at the speed of 180 RPM. The tests were conducted for 20 hours (4 hours for each interval) which is equivalent to 4000 km of travel distance at ambient temperature. After each trial the test samples were thoroughly cleaned in acetone and weighed. The weight loss was converted to volume loss for each sample. Surface Roughness Test was done by using Alicona tester machine to observe the surface structure of the samples before and after slurry erosion test.

Microstructure and Scanning Electron Microscopy (SEM) observations

The specimens for microstructure observations were prepared according to standard metallographic specimens preparations. Specimens were cut using special cutter, hot mounted, grinded and polished before undergo observation process using Olympus B X 41M optical microscope. The specimens were chemical etched with 2 % Nital before the observations. The fractured surfaces of broken tensile and impact specimens were then observed through Scanning Electron Microscopy observations using INCAP entaFETX3 – SEM equipment.

Results and Discussions

Microstructure Observations

Based on the microstructures shown in Figure 2, the differences in the microstructure of the samples of different austempering time can be observed. In Figure 2(a), it can be seen that the graphite found in the material is shaped like a nodule. Due to the nodular or spherical shape of the graphite in the as cast ductile iron, its molecules have lesser discontinuities and give rise to the property of ductility. Graphite nodule is the stable form of pure carbon in cast iron and it plays a significant role in determining the mechanical properties of ductile irons. Figure 2(b) indicates the specimen austempered at 350 ºC for 30 minutes shows the structure that consists of retained austenite, ferrite, and some amount of martensite. In the austempering process done on the alloyed ductile iron, the samples were hold at Austenitizing temperature of 900 ^{o}C for 1 hour. This process dissolves the carbon in austenite, thus producing retained austenite. Figure 2(c) shows that there is structure of austenite in the form of light shaves. In Figure 2(d), the structure of martensite can be seen through the observation. The martensite will disappear from the microstructure if the austempering time is increased more than 1 hour. Higher austempering temperature yields a plate-like morphology of bainitic ferrite with the higher amount of retained austenite. It is obvious that at austempering time longer than 1 hour, no presence of martensite could be detected and the structure only consists of bainitic ferrite and retained austenite. This may be explained by the fact that at short austempering time the carbon content is insufficient to retain austenite stable and, therefore, it transforms to martensite. However, at longer austempering times carbon enrichment was sufficient to stabilize austenite even after air-cooling.

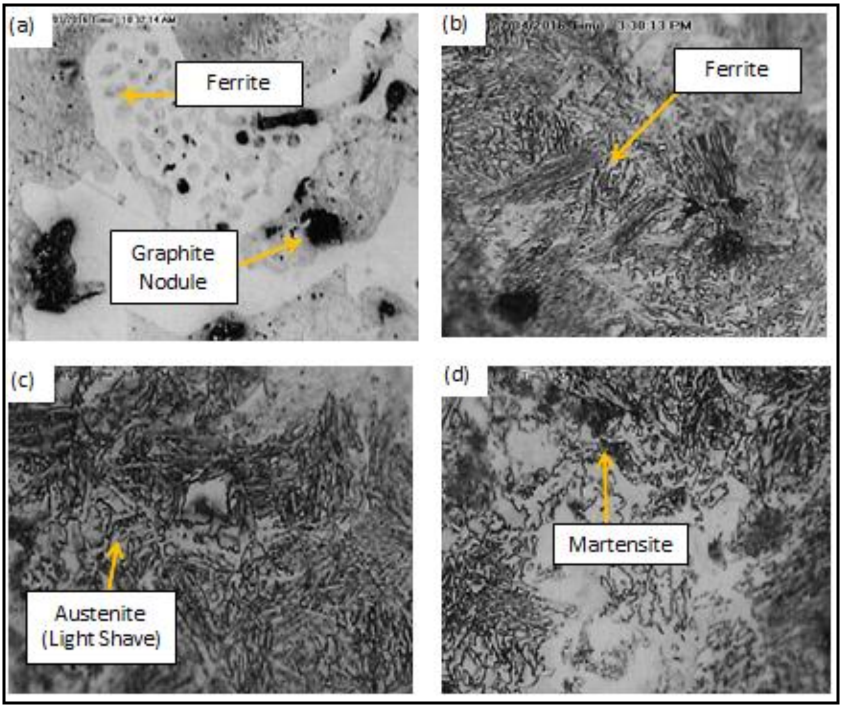

Figure 2. Microstructure Observation (a) As-Cast 0.16 % Cr + 1.32 % Ni DI (b) 0.16 % Cr + 1.32 % Ni DI (30 minutes) (c) 0.16 % Cr + 1.32 % Ni DI (40 minutes) (d) 0.16 % Cr + 1.32 % Ni DI (60 minutes)

Tensile

The results for the tensile test of all types of specimens show the tensile strength and elongations values are shown in Figure 3. Addition of Chromium and nickel was found to have lower tensile strength at the same time decrease the ductility of the material compared to conventional ductile iron specimens. This was attributed to the result of fully pearlitic content in the material [8]. The results also indicated that 0.16 % Cr- 1.32 % Ni ADI (1h) gives the optimum tensile strength compared to other types of specimens due to the presence of bainitic ferrite and retained austenite. However this trend is contrary to Eric et al [9], the high tensile strength shown by the material was attributed by the low amount of retained austenite. The tensile strength value after austempering process was 788.5 MPa, which higher than the values obtained in this study. The percentage elongation also behaved in the same way as that of tensile strength, which is the average value of the elongation of the austempered ductile iron is higher than the elongation of the as cast ductile iron.

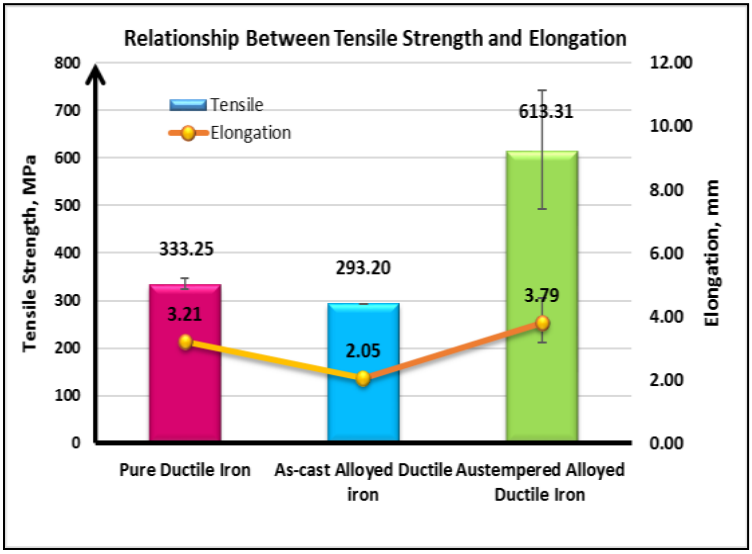

Figure 3. Relationship between tensile Strength for 3 different types of material

Impact Toughness

It was observed that addition of alloying elements resulted in favorable effect to the impact toughness properties as 25 % increment as compared to DI samples (shown in Figure 4). Austempering the samples at 1 hour holding times improve 50 % of the impact compared to DI samples, given by fine bainitic ferrite and retained austenite structures. Fine structure of ferrite platelets and the low amount of retained austenite contribute to this high strength and toughness [10]. This showed that the as-cast Ductile Iron is more brittle then the austempered ductile iron. When the sample was struck by the pendulum of the impact test machine, the tougher material which is the austempered ductile iron can absorbed a significant amount of energy while the less tough material which is the as cast ductile iron was only able to absorb relatively little energy before fracture. As the temperature of austempering process used is high (350 ºC), it changes the microstructure from martensite to cementite. Thus, the structure of the austempered ductile iron is much softer and ductile therefore it can absorb more energy of the impact. According to [11], the additions of 2 % Vanadium into ductile iron slightly reinforce the dynamic impact resistance through increases the impact properties by 43 % increment. The increment trend also showed by when an amount of Cobalt is added [12] where increment value is noted about 40 %. Nevertheless, the impact properties otherwise is significantly decrease as Nickel element is been added [12].

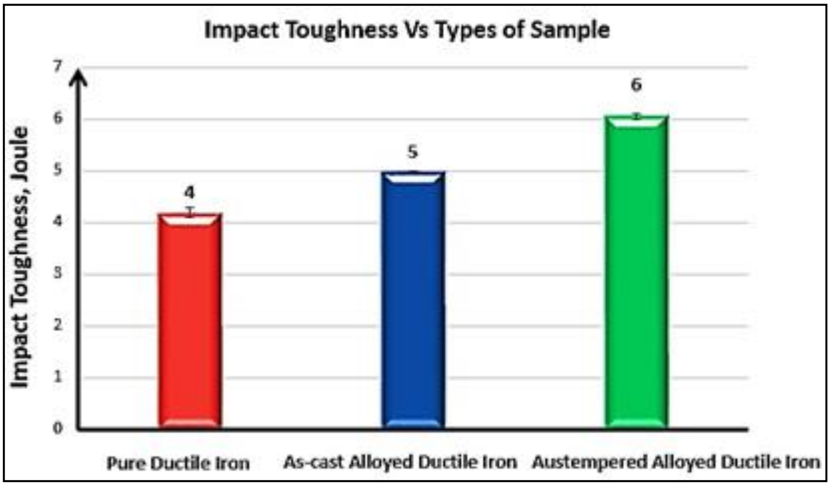

Figure 4. The impact toughness versus different types of materials

Hardness

Figure 5 indicates the average results of Vickers hardness test (HV) for as-cast sample of DI, Cr-Ni Alloyed DI and Cr-Ni Alloyed ADI. Hardness significantly increases after the sample has been austempered. The pearlitic structure revealed after heat treatment process improves the hardness rather than ferritic-pearlitic structure shown in as-cast. It occurred due to fine pearlite structure achieved as ductile iron treated and thus assisting in solid solution hardening [13]. The such properties shows due to the addition of alloying element itself contributed in accelerates and stabilizes the pearlite formation thus increasing the hardness result compared to conventional ductile iron [14]. In addition the highest hardness value shown by the heat treated sample in this study is almost consistent as the hardness value shown by ADI [13]. Sample of Cr-Ni alloyed DI austempered at 300 ˚C is higher than as-cast Cr-Ni alloyed DI with 49 % increment. However, high hardness values are not convenient if the material going to be under impact due to fragility. Otherwise, it is recommended as the material going to be under a friction force.

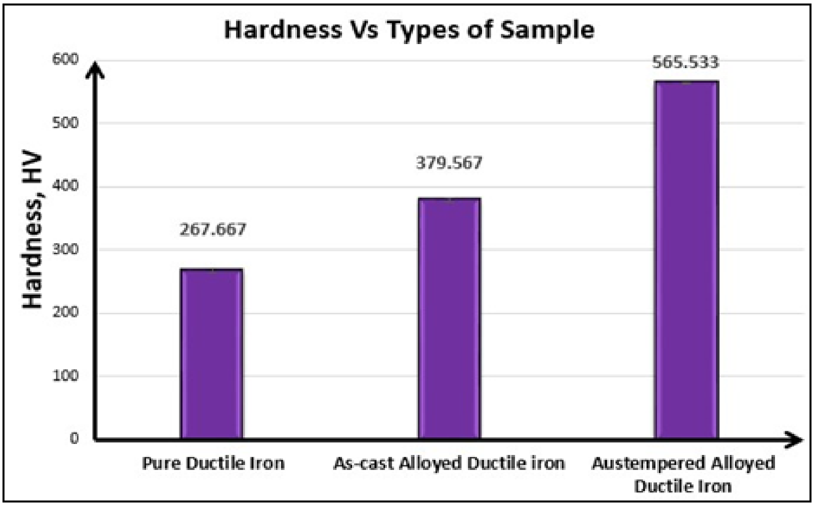

Figure 5**.** Hardness versus different types of materials

Slurry Erosion Wear Test

Based on Figure 6 (a), the graph illustrates the decreasing trend of the weight of both of the samples of as cast alloyed ductile iron and austempered alloyed ductile iron. This shows that the samples undergo erosion process during the wear test. In Figure 6 (b), the weight losses of both the samples against time were shown. Referring to the figure, for the as cast alloyed ductile iron, the value of the weight loss for the first three intervals, 4 ,8 and 12 hours increases quite drastically before declining for the next two intervals. As for the austempered alloyed ductile iron, the weight loss of the samples has a quite constant pattern except for the first interval where the value of the weight loss is quite high compared to other intervals. T.R. Uma et. al [15] also reported that the enhanced wear resistance of ADI is due to the ability of carbon stabilized austenite to strain hardened and in some cases undergo transformation to martensite. Like strength, ductility, toughness and work hardening capacity. Generally, hardness is considered as the most important property which affects the erosion behaviour of material [16]. Several studies [17][15] concluded that increase in hardness alone is not sufficient to improve the erosion resistance of steels. However, the combination with other properties such as ductility, toughness, work hardening and impact strength provided significant effect on the erosion behaviour. Hutchings [18] [18] also proposed that high hardness with some amount of ductility offers better erosion resistance. A.Kumar et.al [19] proposed that ductility of the steels had a significant effect on erosion resistance. A.Kumar et.al [19] also suggested that initially erosion resistance increases with increase in hardness, and as hardness reaches its critical value, further increase in hardness leads to tendency of embrittlement, which causes decrease in erosion resistance.

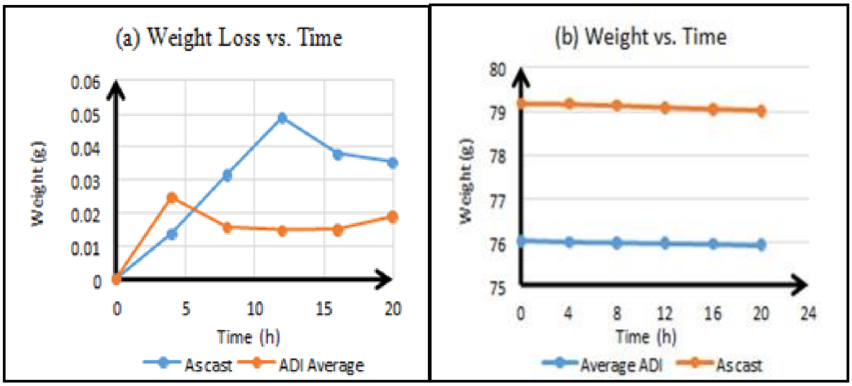

Figure 6. Slurry Erosion Test of As Cast Alloyed DI and Austempered Alloyed DI (a) Weight Loss vs Time (b) Weight vs Time

Scanning Electron Microscope (S.E.M) Observations

Based on the observations on the S.E.M image in Figure 7 (a), Figure 7(b), Figure 8, and Figure 9 it was discovered that the topography of the all the samples shows that there are signs of defects in the structure of the samples. Figure 7(a) and 7(b) shows that there are ductile fracture and cleavage fracture in the structure of the samples. These defects may lead to decreasing value of the strength of the sample as the fracture may reduce the ductility of the material [20]. Referring to Figure 8, it shows the existence of cleavage facets of ferrite in the structure of the specimen that undergo impact test. This may be the reason of the low impact toughness value of the austempered alloyed ductile iron sample obtained from the test. Based on Figure 9, it can be seen that there are also cleavage fractures in the structure of the material that may cause difference in the weight loss of the sample.

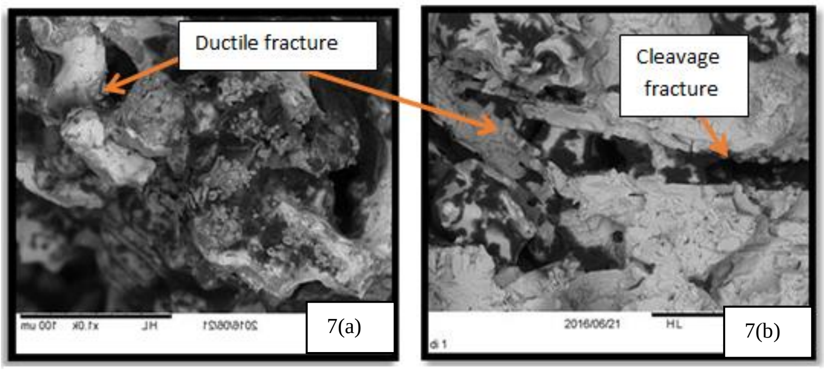

Figure 7. S.E.M image of fracture topography at magnification of 1000x (a) As cast ductile iron (tensile) (b) Austempered Alloyed DI (tensile)

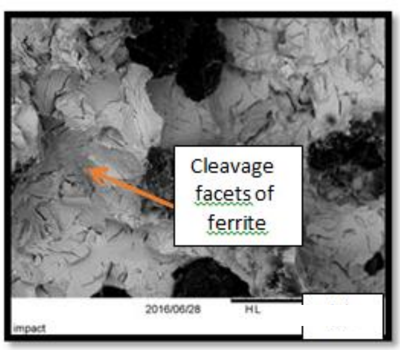

Figure 8. S.E.M image of fracture topography at magnification of 1000x for Austempered Alloyed DI

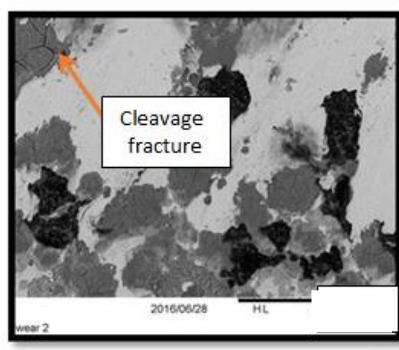

Figure 9. S.E.M image of fracture topography at magnification of 1000x for Austempered Alloyed DI (wear)

Roughness Test

Table 2 shows the value of the average roughness, Ra of each specimen which were the as cast ductile iron and alloyed ductile iron. It shows decreasing value after the Slurry Erosion test was done on the alloyed ductile iron, however different with the value of Ra for the as-cast specimen which shows that the Ra value increased. Theoretically, roughness is often closely related to the friction and wear properties of a surface. A surface with a large Ra value will usually have high friction and wear quickly. Based on the data, Ra value of As-cast specimen for increased from 1.1191 µm to 1.8053 µm which means the surfaces easily wear. Meanwhile, Ra values for alloyed ductile iron decreased from 2.0771 µm to 1.7498 µm which means the surface was hard to wear after underwent Slurry Erosion test. The roughness of each specimen probably effected form Slurry Erosion test as it were immersed in the slurry container that contained of water and sand and the specimen were rotated at constant speed in order to complete the 20000 km travel distance. Thus, the Ra increased as the specimen surface becomes rougher.

Table 2: Surface Roughness, Ra Value of as Cast Alloyed DI and Austempered Alloyed Ductile Iron

Sample	Surface Roughness, Ra (µm)	
	Before Wear	After Wear
As cast	1.1191	1.8053
Alloyed ADI	2.0771	1.7498

Conclusion

This study focused on the mechanical properties and physical properties of alloyed ductile iron (0.16 % Cr + 1.32 % Ni) which undergo heat treatment of austenitizing at 900 ^{o}C for 1 hour, then austempered by quenching in the salt bath at the temperature of 350 ^{o}C for 40 minutes. The samples then undergo several test such as microstructure observation, tensile test, impact test, wear test, surface roughness test, hardness test and scanning electron microscopy (S.E.M). Based on the analysis of the results obtained from the study, it can be concluded that:

I. The hardness value of the austempered ductile iron is higher than the hardness value of the as- cast ductile iron. This was due to the austempering process of 350 ºC for 40 minutes that produces retained austenite which improve the hardness of the material improved. The tensile strength value for the austempered ductile iron improved by nearly 200 % when compared to as cast ductile iron. The as cast Ductile Iron has lower toughness than austempered ductile iron.

II. Microstructure observation of the materials shows the formation of spheroidal graphite nodule. The other structure observed consists of retained austenite, ferrite, and some amount of martensite.

III. The fracture topography shows existence of ductile fracture, cleavage fracture and cleavage faces of ferrite in the sample after tensile, impact and wear test.

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