Effects of Magnesium Variation and Heat Treatment on Mechanical and Micro-Structural Properties of Ductile Cast Iron

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Abstract - The effects of magnesium variation and austempering heat treatment on mechanical and microstructural properties of ductile iron produced using the rotary furnace were investigated. Varied quantity of magnesium-ferrosilicon in the range of 0.03 % to 0.06 % were used as nodulirizer to treat 4 kg mass of molten metal per ladle by sandwich process and poured into sand mould. Mechanical test (tensile, hardness, fatigue, impact and wear) and micro-structural examinations were carried out on the four samples produced. Samples C and D of 0.056% and 0.061% magnesium showed an improvement in their micro-structural properties due to the presence of more graphite nodules. Hence, they were observed to have exhibited better tensile strength of 598.07MPa and 609.03MPa. The fatigue strength also increased to 501.91MPa and 509.27MPa respectively. These two samples were further subjected to austempering heat treatment by heating to 850ºC for austenitization and soaked for homogenization for one hour at the temperature before quickly transferred into a salt bath of 50 % NaNO3 : 50 % NaCl maintained at 3600C and quenched for transformation for 1 hour before finally air cooled. Mechanical tests and micro-structural examinations were thereafter carried out. Sample C had an outstanding increase in tensile strength, from 598.07MPa to 891.22MPa, while specimen D increased from 609.03MPa to 898.76MPa. The results of abrasion test indicated that samples C and D had abrasion resistance increase from $2.20\times10^{11}m^2$ and $2.39\times10^{11}m^2$ to $2.35\times10^{11}m^2$ and 2.68×10^{11} m^2 respectively after austempering. There were also relative increase in fatigue resistance and impact toughness for the two samples but with relative reduction in hardness from 47.7 to 44.2 and 50.3 to 47.4.

Keywords - Magnesium, Flaring, Nodules, Austempering, Mechanical properties, Microstructures

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1 Introduction

Ductile iron is also known as spheroidal graphite and nodular iron is made by treating the liquid cast iron with special "spheroidising" elements to promote the precipitation of graphite in the form of spheroids rather than as interconnected flakes (Pierre-Marie, 2011). Alkaline earth metals that include magnesium and calcium and the rare earth metals such as cerium, lanthanum, and yttrium have been proved to be these special elements (Bockus and Dobrovolskis, 2006). Nodularization process is the single most important step in production of ductile iron and production of quality castings is determined by how well a foundry can precisely control the level of magnesium in their melt (Sesham, 1998). After rapid growth for more than 50 years, ductile iron has become one of the most important engineering materials and gray iron/ steel castings are being replaced by ductile iron castings with similar mechanical properties maintained (Pierre-Marie, 2011).

There are many different treatment methods of spheroidization to produce ductile iron. All of which involve the introduction of the spheroidizing agent in some form; either alone or in combination with other rare earth metals. Successful production of high quality ductile iron depends on the appropriate treatment of the melt and avoidance of fading in order to achieve satisfactory nodularization (Adewuyi, 2002). Many researches have been carried out on ductile iron produced using induction furnace because it is easy to calculate and control the charge parameters, thus making sure the desired composition of the casts are obtained (Oyetunji, 2007).

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However, researches are being conducted in recent times with the use of rotary furnace which has been discovered to reduce the overall cost of production, encourage small scale production and also the problem of epileptic power supply will be minimized (Adewoye, 2005). The present work is therefore aimed at determining the actual percentage of the magnesium required to give optimum mechanical and micro-structural properties of as-cast and austempered ductile cast iron and to determine what quantity of charge materials will compensate or take care of the reactions that may be evolved during melting and casting processes using diesel fired rotary furnace. The work also seeks to employ the use of recycle graphite in discarded battery cells to be used with other locally sourced materials thereby reducing cost.

2 Experimental Procedure

Wooden cylindrical patterns of diameter 20 x 230 mm length were produced using 1.5 m lathe machine. The down sprue of diameter 40 mm was tapered to diameter 30 mm with a length of 200 mm and overflow of diameter 80 x 220 mm were also produced. Both the pattern and sprue were designed with a contraction allowance of 1.5% in line with the practice of Alasoluyi et al (2013). The moulding sand used was prepared by adding 12.5% bentonite as binder with 8 % water into silica sand. Sixty kilogram (60kg) of grey cast iron scraps of 3.8 %C, 2.19 %Si, and 0.123 %S and 0.3 % P with 2 kg of graphite (recarburizer of 66 % grade). 0.096 kg FeSiMg of 4.8-5.2 %Mg was used as nodularizer and 0.25 kg calcium carbide were charged into an oil-fired rotary furnace of 100 kg capacity.

The furnace was first pre-heated for one hour before the materials were charged. To ensure even distribution of graphite charged, the charging was done in alternating layer as earlier practiced by Agarwal, et al, (2007). This was heated to a tapping temperature of 1506 ºC, then 0.25 kg of pulverized calcium carbide was added. This desulphurised the sulphur in the melt before it was tapped into already pre-heated ladle built to conform to standard sandwich process of adding magnesium into molten metal (BCIRA, 1998).

To obtain the quantity of graphite that will give about 2 %C in the melt inside the rotary furnace, equation 1 was used:

$$\text{Quantity Required} = \frac{\%\ \text{Element in the Melt x Total Mass}}{\%\ \text{Element of Ferro Alloy}} \quad (1)$$

Samples C and D were austempered using muffle furnaces. They were austenitized at 850 ºC, held at this temperature for 1 hour and austempered in a salt bath containing solution of 50% $NaNO_3$ and 50% NaCl at 360ºC for one hour before finally air cooled to room temperature. Both the as-cast and austempered samples were then subjected to mechanical tests and micro-structural examinations.

Metallographic samples obtained from the cylindrical bars produced were prepared with Buehler metasev 2000 grinder and polisher. Grinding was done with the use of different grinding papers of 60, 120, 400, and 600 grits one after the other. After grinding, polishing was also done using 800, 1000 and 1200 grits of emery papers. Final polishing was achieved on polishing cloth with diamond polish suspension of 3 microns. They were etched using 2.5% Nital solution with etching time of 12 seconds. Photomicrographs were obtained using AP2000MTI Metallurgical microscope with Daheng digital camera having a maximum magnification of X800.

Rockwell hardness tester was used to determine the hardness of every sample produced. The tensile specimens were machined from each of the cylindrical cast bars to conform to Instron Universal Tester specification. Instron Universal Tester is a computerized machine which reads all the data, plotted the graph as tensile strain takes place. This test was used to establish tensile strength and percentage elongation. Impact samples were also machined 60mm length and v-notched at the center at an angle of 45o with 2mm depth. Avery Denison impact testing machine was used to carry out the Charpy impact test. The specimens for this test were machined to 60 mm length and v-notched at the center at an angle of 45o with 2mm depth. The test specimen for the fatigue tests were also machined to specification. Due to the rotation, the upper surface of the test piece was subjected to tension, whereas the lower surface experienced compression (Kenawy, et al 2001). The rotation of the surfaces continued till failure occurred. The tests were performed at various cyclic stresses. Thus a graph was obtained between cyclic stresses and number of cycles. The fatigue limit was then read from the plot of S-N curves obtained. High stress abrasion testing was done by using the pin abrasion method per ASTM G132-96 while low stress abrasion was conducted using the wet sand/rubber wheel test per ASTM G105-89 (ASTM Standard G 132—96 and G105-89). This was done by loading the cylindrical test sample against a rotating wheel and sand of 800 grits was deposited on it at a controlled flow rate between them. The wheel was rotated in the direction of the flow of the sand thereby causing the surface of the test sample to peel off. The machine was set to 600 revolutions per minute and the test was conducted in 300 seconds. The initial and the final weight of the test samples were recorded. All the mass loss data from the abrasion tests were converted to volume loss by dividing them by the material density so that comparison of the results can be made to determine which of the samples had the greater loss in mass.

3 Results of Analyses

3.1 Chemical Analyses

The results of the chemical composition of the four ductile iron produced are contained in Table 1.

Table 1: Chemical Composition of Cast Samples A – D

Sample	A	B	C	D
C	3.7861	3.6661	3.5482	3.4921
Si	2.3321	2.3428	2.3361	2.3671
Mn	0.0891	0.0894	0.0868	0.0879
Mg	0.0322	0.0452	0.0561	0.0614
S	0.0831	0.0721	0.0451	0.0437
P	0.0437	0.0626	0.0874	0.0869
Al	0.0078	0.0063	0.0059	0.0074
Cr	0.0915	0.0921	0.0935	0.0963
Cu	0.0626	0.0498	0.0332	0.0621
Fe	94.01123	94.01332	94.22823	94.30123

3.2 Micro-structural Analyses

Plates I-IV show the results of photomicrographs of the un-heat treated samples A to D, while Plates V and VI are for the samples C*and D* austempered respectively.

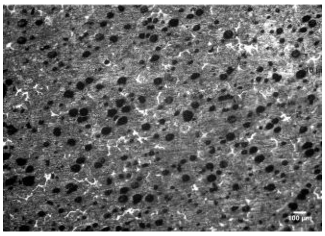

Plate I: Microstructure of sample A showing graphite nodules in ferrite matrix with 2% Nital etched (X100)

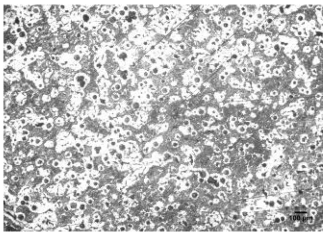

Plate II: Microstructure of sample B showing some surrounded by ferrite and pearlite etched (X100)

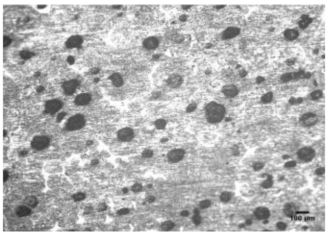

Plate III: Microstructure of sample D showing perfect graphite nodules in ferrite matrix with 2 % Nital etched for seconds

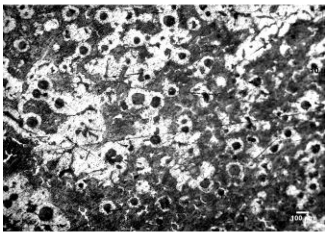

Plate IV: Microstructure of sample D showing spiky nodules and undissolved flakes with 2 % Nital etched for 10 seconds

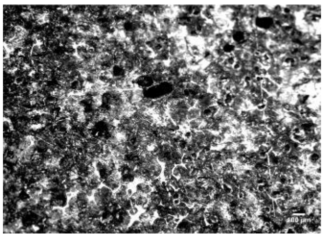

Plate V: Microstructure of sample C Austempered showing Ausferrite and pearlite matrix with undissolved ferrite. 2% Nital etched for 10 s)

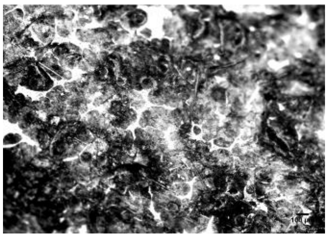

Plate VI: Microstructure of sample D Austempered showing Ausferrite pearlite matrix with undissolved ferrite. 2% Nital etched for 10 s)

3.3 Mechanical Tests Results

The summary of the results of the tensile tests, impact tests, abrasion resistance tests, fatigue tests and hardness tests results for as-cast and austempered samples are as contained in Tables 2 and 3 while Figures 3 to 8 explained the relationship among the variation in % Mg added with the mechanical properties of both as-cast and austempered cast samples.

Table 2: Result of Mechanical Tests of As-Cast Samples

Sample	A	B	C	D
% Mg	0.029	0.038	0.056	0.061
Hardness (HRA)	48.9	48.3	47.7	50.3
Impact Strength (J)	8.42	9.67	12.83	13.41
% Elongation	14.89	16.23	26.74	28.51
Tensile Value (MPa)	392.94	402.52	598.07	609.03
Fatigue Strength (MPa)	404.72	412.76	501.91	509.27
Abrasion Resistance (x1011 m2)	2.01	2.04	2.20	2.39

Table 3: Result of Mechanical Tests of Austempered Samples

Sample	C*	D*
% Mg	0.056	0.061
Hardness (HRA)	44.2	47.4
Impact Strength (J)	12.44	13.95
% Elongation	27.74	29.51
Tensile Value (MPa)	812.21	898.54
Fatigue Strength (MPa)	521.54	532.85
Abrasion Resistance (x1011 m2)	2.35	2.68

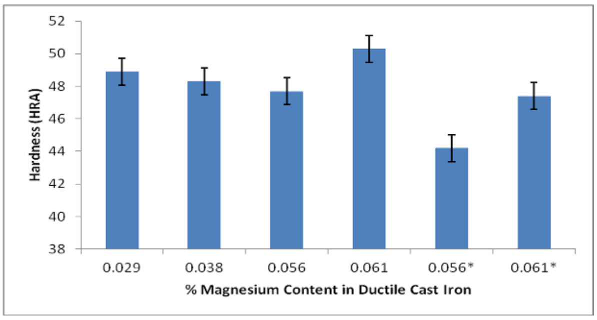

Figure 3: Effect of Mg Variation on the Hardness of As Cast and Austempered Samples

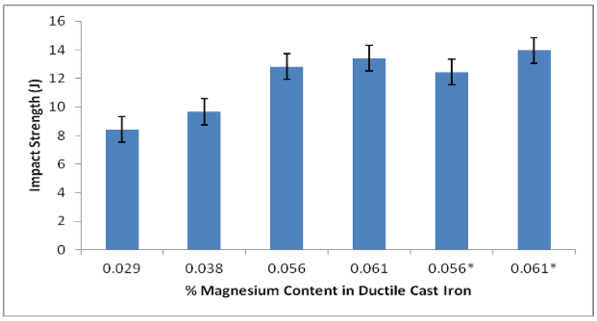

Figure 4: Effect of Mg Variation on the Impact Strength of As-Cast and Austempered Samples

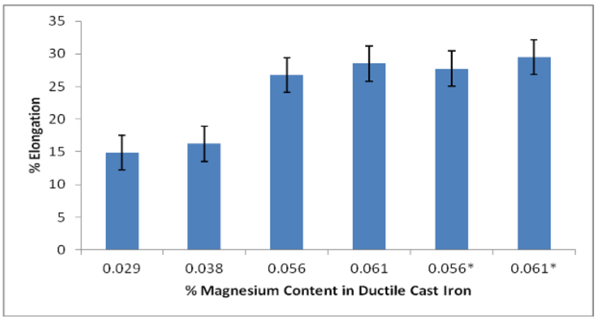

Figure 5: Effect of Mg Variation on the % Elongation of As-Cast and Austempered Samples

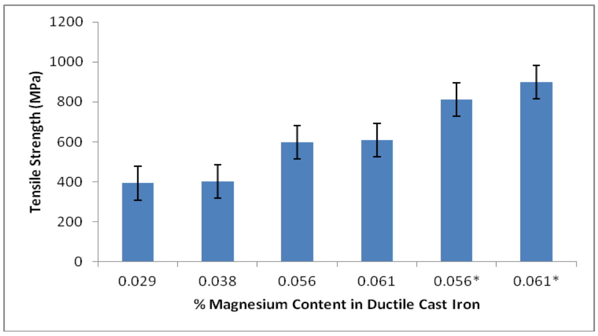

Figure 6: Effect of Mg Variation on the Tensile Strength of As-Cast and Austempered Samples

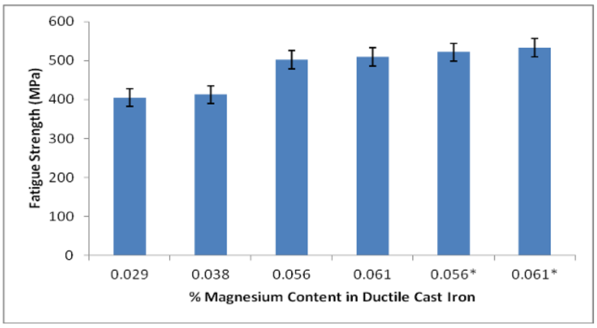

Figure 7: Effect of Mg Variation on the Fatigue Strength of As-Cast and Austempered Samples

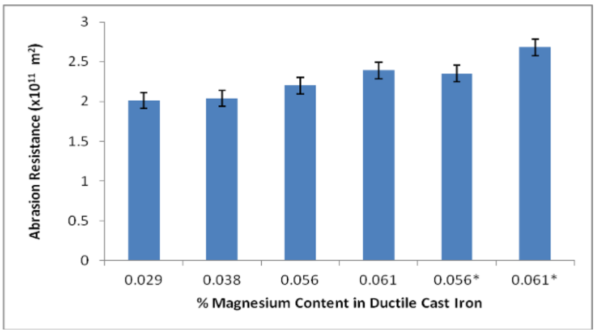

Figure 8: Effect of Mg Variation on the Abrasion Resistance of As-Cast and Austempered Samples

4 Discussion of Results

4.1 Effect on Microstructure

The study revealed that there was a reduction in the %C of the samples produced compared to the 3.8%C in the base metal despite addition of 2kg graphite of 66% grade, this may have been due to the loss in the carbon. There was carbon oxidation which was due to the mode of operation of rotary furnace in which the fuel, charges and the products of combustion are all in contact with each other. This can be used to determine the efficiency of the rotary furnace. However, the amount of carbon loss through oxidation is a function of time (in minutes) spent during melting (Ibrahim et al, 2008). The more the melt stay in the furnace, the more the carbon loss.

American Foundrymen's Society (2005) had reported that the rate of magnesium loss from the molten metal to be 0.001 % per minute at 1482ºC. The results of spectrometric analysis in Table 1 show that the magnesium loss was pronounced. This was due to flaring and magnesium fumes formed despite sandwich method of magnesium addition used. The reaction of magnesium with sulphur in the molten metal could also account for the loss in the magnesium. Sulphur is more reactive with magnesium than iron and the process usually favours the removal of sulphur before treatment. This was why calcium carbide was introduced into the melt six minutes before the tapping. It has also been reported that the spectrographic analysis widely used to determine the chemical composition in the cast could not indicate the exact magnesium that is responsible for causing graphite spheroids from the one that reacted to form MgS, MgO or MgSiO3 (Itofuji, 1999).

From the study, it was observed that sample A (0.0322 %Mg) and B (0.0452 %Mg) have fewer or sparse number of nodules both of which are in the matrix of pearlite despite the quantity of nodulirizer added as observed in Plates I and II. This was because the magnesium deoxidized and desulphurised the melt and the formation of carbide eutectic could not be promoted. This suggested that the base iron may not have been properly controlled; hence, excessive oxygen and/or sulphur consumed the magnesium leaving an insufficient amount to spheroidize the graphite. The amount of sulphur in the melt must be reduced to 0.02 % before the remaining magnesium could enable the precipitation of carbon to become graphite nodules. Magnesium will have to react with sulphur first and reduce its percentage to less than 0.02 %. It is the remaining magnesium that will then combine with the carbon to form nodule and this will impart its effects on the microstructure and hence, significantly influence the mechanical properties ((Imasogie et al, 2001; and Omole and Oyetunji, 2007). Sample C that contained 0.0561 %Mg had an increased volume of nodules in an entirely pearlitic matrix with traces of ferrite as shown in Plate III while sample D containing 0.0614 had a ferritic matrix more than that of C with lesser volume of nodules also compared to C as observed in Plate IV.

4.2 Effect on Mechanical Properties

The presence of graphite nodules usually improve wear resistance as the nodules enhance heat transfer and helps to lubricate the sliding wear surfaces. The dispersed graphite nodules in the ferrite matrix of as-cast ductile iron of samples C and D afford some appreciable resistance to frictional wear. These are indicated in Table 2 and Figure 8 where the values foe samples A and b were 2.01 x 1011m2 and 2.04 x 1011m2 and increased to 2.20 x 1011m2 and 2.39 x 1011m2 respectively for samples C and D. This was improved upon in samples C* and D* to 2.35 x 1011m2 and 2.68 x 1011m2 that were austempered. The volume of the nodules formed as influenced by the %Mg in samples C and D are responsible for the substantial increase in the tensile strength of 598.07MPa and 609.03MPa respectively. These also accounted for the improvement in the fatigue strength of 501.91MPa and 509.27MPa of C and D compared to 404.72MPa and 412.76MPa for samples A and B respectively. These are as shown in Table 2; Figures 6 and 7. It has been established that the presence of nodules act as hindrances to the propagation of crack thereby increasing the strength of the material. The same trends were also followed in the observed results of %elongation and impact strength as observed in Figures 4 and 5. Ductile iron grades are characterized by their tensile strength, yield strength, and elongation, but these properties are not alone always used in determining the suitability of their use in a specific application (Bockus and Dobrovolskis, 2006).

The austempered sample C* and D* contained few nodules with a mixture of ausferrite and undissolved ferrite matrix, ferrite being more in C* than D*. This is the responsible factor for the high strength of 812.21MPa recorded in sample C. The result of the experiment indicated that the impact strength of as cast samples as shown in table 2 increased as the percentage of magnesium increased. There was also an increase in the impact strength of the heat-treated samples. However, toughness of ductile iron depends on ferrite/pearlite ratio (Omole and Oyetunji, 2007) and this can be observed in the results of as-cast samples when compared to the austempered ones in table 2 and 3. The hardness values of as cast samples were observed to increase with increase in graphite addition as shown in Table 2. The hardness values of as-cast samples shown in Figure 3 as measured on HRA scale of 60 kg load ranged between 48 and 53. The values reduced from 47.7 to 44.2 and 50.3 to 47.4 in as-cast and austempered samples C* and D* respectively.

5 Conclusion

The following conclusions were therefore drawn from the study:
(i) The sample D that contains 0.061% of magnesium gave the maximum tensile strength of 609.04MPa. It also gave the maximum values of the properties evaluated, hence, optimum mechanical and micro-structural properties. The magnesium loss were due to flaring, desulphurization and oxidation
(ii) The rotary furnace process route of ductile iron production lead to direct contact between the oxidizing flame and charge which lead to carbon loss. The longer the melt stayed in the furnace the more the loss of carbon.
(iii) The austempering heat treatment carried out improved the mechanical properties as shown in Tables 2 and 3; and Figures 3-8. These were at the expense of the hardness.
(iv) The abrasion resistance result of samples C and D show a remarkable improvement which is due to the increase in strength as a result of the increased volume of graphite nodules and pearlite.

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