



Impact of Sandwich Process Master Alloy Proportions on Nodule Precipitation and Properties of Thin Wall Ductile Iron

Ezenwanyi Fidelia OCHULOR¹, Eugenia Obiageli OBIDIEGWU², Sunday Chukwujekwu ANAH³, John Obioma UGBOAJA⁴

^{1,2,3,4}Department of Metallurgical and Materials Engineering, University of Lagos, Akoka, Lagos State, NIGERIA
ochulor@unilag.edu.ng / eobidiegwu@unilag.edu.ng / Anahchukwujekwu@gmail.com / jon_obioma@yahoo.com

Corresponding Author: ochulor@unilag.edu.ng

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Abstract: Melt treatment via introduction of magnesium (Mg) master alloy during casting of thin wall ductile iron (TWDI), is plagued with a number of challenges such as excessive oxidation, fume and slag formation, carbide precipitation, shrinkage tendency as well as its fading tendency arising from peculiar properties of Mg with respect to molten iron. In this study, variation of the weight percent of master alloy was investigated to determine effects on magnesium recovered and consequently, on nodularity, nodule count and mechanical properties of the cast TWDI. Samples were produced using sandwich method by treating melt of suitable composition with Mg master alloy varied in proportion from 0, 1, 2, 3, 4 and 5 wt.%, two stage inoculation process was carried out. Microstructure examination was carried out according to ASTM E407 and 247 specifications: nodularity and matrix type present within the cast TWDI samples. Magnesium content prior to casting was determined via spectrometric analysis, hardness test was carried out according to ASTM E10 using the brinell scale. The Ultimate tensile strength (UTS) was obtained in accordance with ASTM E8 standards. Results obtained from the microstructure examination, showed presence of ferrite and pearlite matrix within the microstructure of all the samples, 3 wt. % Mg master alloy treated TWDI sample possessed pearlitic phase dominated microstructure. Percent nodularity and nodule counts showed optimum values of 94.1%, 821 and 92.6%, 735 nodules/mm² for 2% and 3% alloy treated samples respectively. Matrix type consisted of ferrite and pearlite phases observed for MA2 and MA3 samples, whereas carbide precipitations were included in all other samples. The control sample (MA0) which was not magnesium treated showed graphite flakes. This study has shown that increased proportions of magnesium master alloy did not lead to better nodule characteristics instead carbide precipitates and decline of mechanical properties were observed.

Keywords: Thin wall Ductile Iron (TWDI), melt treatment, Mg master alloy, nodules and carbide precipitates.

1. INTRODUCTION

Ductile iron (DI) is a family of cast iron materials having a wide range of mechanical properties obtained through proper microstructural control. It consists of dispersed graphite spheroids in an iron matrix, which may be totally ferritic or pearlitic in the ductile grades, martensitic in the strong and hard grades, or bainitic in the strong and tough grades [1]. The crucial need to remain a material of choice for automotive application has necessitated thickness reduction in cast DI parts leading to TWDI which are DIs having thickness range of less than 5mm [2], its production has opened up areas where DI could be used to replace aluminium alloys for light weight automotive parts [3, 4]. Melt treatment in DI is necessary for the production of requisite microstructure and mechanical properties, inoculation and nodularization treatments are the two crucial melt treatments conducted in casting DI. In the inoculation process the melt is treated with ferrosilicon to initiate heterogeneous sites for graphite nucleation, this process is carried out at least twice to improve the yield of the process whereas in the nodularization treatment, pure magnesium or magnesium master alloy is adopted to tie up Sulphur (S) and Oxygen (O) allowing graphite to grow as nodules or spheres instead of flakes, which is the characteristic shape of graphite particles formed in DI. Increased demand and production of DI in recent years implied the need to find consistent and cost efficient production processes. Two routes evolved in a bid to solve this need. One focused on introducing low amounts of pure Mg or high Mg containing master alloys. The other looked at developing a cost efficient way through introducing dilute Mg which alloys a calm reaction and reduction in the negative impact of high residual Mg content on shrinkage tendency and carbide formation. Treatment methods focused on minimizing the addition of Mg such as ladle and in mould treatments [5].

The simple ladle Mg master alloys offered the advantage of adding Mg in a quieter reaction with higher recovery rates than pure Mg addition method. The researchers in [6] investigated the effects of magnesium variation and heat treatment on mechanical and microstructural properties of DI, from the study, it was observed that sample A with 0.0322 % Mg and B with 0.0452 % Mg have fewer or sparse number of nodules both of which are in the matrix of pearlite despite the higher

quantity of nodularizer added. This resulted from de-oxidation and desulphurisation of the melt by the magnesium and the formation of carbide eutectic. 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 nodularize the graphite. Sample C containing 0.0561 % Mg had an increased volume of nodules in an entirely pearlitic matrix with traces of ferrite, while sample D containing 0.0614 had a larger volume of the ferrite matrix than that of sample C. Magnesium master alloys can be classified as light and heavy, depending on their density relative to liquid iron [7]. Depending on the characteristics of each master alloy, different treatment methods are used. The magnesium content is a very important characteristic of master alloys. With increasing magnesium content, magnesium recovery in liquid iron decreases while pyro effect increases. Typical light FeSiMg master alloy contains 3.0 – 10.0% Mg, 43 – 48 percent Si, 0.8 – 2.0% Ca and up to 2.5% Ce. Nickel- or copper-based master alloys may contain from 5 – 15% Mg, with rest being Ni or Cu. In some cases, the nickel can be replaced by 32 – 36% Fe or 26 – 33 % Si. Nickel or copper-based master alloys have a higher density than liquid iron. This eliminates the need for special mixing to prevent the master alloy from floating on the liquid iron surface. Nickel and copper acts as strong pearlite stabilizer and light graphitizes, minimizing variations in mechanical properties between thin and thick sections of castings. Light master alloys require the use of appropriate methods for improving Mg recovery by keeping the treatment alloy below the liquid iron surface until it is fully dissolved, thus minimizing Mg losses and reducing pyro effect. Among these methods, the most widely used are in-ladle, in-mould, and flow through process [8]. The sandwich process adopted in many Foundries is an improved modification of the open ladle process. In this process, the magnesium ferrosilicon is introduced into pocket built into the ladle and is covered with either steel punching or ferrosilicon. The cover material acts as a physical barrier between the nodulant and incoming molten iron which delays reaction time therefore increases efficiency. The major advantages of this method are simplicity, flexibility and low cost. The disadvantages include the necessity of using low sulphur base iron, unstable magnesium recovery and relatively high pyro effect [9]. In the study of [10], the researcher investigated the solidification, processing and properties of DI and concluded that the least residual magnesium proportion needed to sustain adequate nodule characteristics in DI parts is 0.03%, however existing literature have scanty information for this requirement for TWDIs, consequently leading to defective TWDIs parts currently cast in many local foundries. These defects can be in the form of shrinkages, white or grey irons, non-nodular TWDI, these impact negatively on properties, leading to rejects and waste of resources. These aforementioned problems have led to the crucial need to establish adequate proportion of master alloy for treatment in Foundries that adopt the Sandwich treatment technique. This study is therefore aimed at establishing the proportion of magnesium master alloy requirements to be adopted for the sandwich treatment process for TWDI parts mainly used for automotive applications.

2. METHODOLOGY

2.1 Materials

The study was carried out at the Foundry Laboratory of Nigerian Machine Tools Limited, Oshogbo, Osun State, Nigeria. Green sand moulds prepared from silica sand particle size was between (250-300 microns) was adopted as the mould material. A wooden pattern of dimensions 3 x 150 x 150 mm was used for mould preparation. Charge materials were melted in 500 kg Induction furnace and consisted of cast iron sleeve scrap, graphite coke, ferrosilicon alloy. Ferrosilicon magnesium (FeSiMg) master alloy was used for nodularization treatment after melting. Chemical compositions of steel scrap, graphite, ferrosilicon and ferrosilicon magnesium master alloy are shown in Tables 1, 2, 3 and 4 respectively.

Table 1: Chemical composition of Steel scrap

Element	C	Mn	P	S	Fe
Wt. %	0.60	0.25	0.03	0.02	99.10

Table 2: Chemical composition of Graphite coke

Constituent	C	Ash	Volatiles
Wt. %	66.00	30.20	3.80

Table 3: Chemical composition of Ferrosilicon Alloy (FeSi)

Element	Si	Al	C	S	P	Fe
Wt. %	70	0.31	0.0032	0.001	0.001	29.68

Table 4: Chemical composition of Ferrosilicon Magnesium Master Alloy (FeSiMg)

Element	Mg	Si	Ca	RE	Al	Fe
Wt. %	7.5	44.5	2.02	0.8	< 0.7	44.47

2.2 Melt treatment and Casting of TWDI samples

The charge materials as shown in Table 5 were heated to melting temperature of 1450^oC, then superheated to 1500^oC to ensure adequate mould filling of the thin section samples, after which 80kg was poured into a preheated treatment ladle for the nodularization treatment adopting the sandwich treatment process. The FeSiMg master alloy was added to the liquid melt

in the treatment ladle in its centre pocket of dimension 227 mm diameter by 114mm depth which was covered with a thin steel sheet to delay the reaction and avoid fading. This process of tapping and treatment was carried out using Mg master alloy with chemical composition shown in Table IV, it was varied from 1, 2, 3, 4 and 5 wt. % for five treatments. Two step inoculation treatment was adopted for each sample, the first being during transfer of the melt from the treatment to pouring ladle and the second being during pouring into the mould cavity where ferrosilicon alloy (FeSi) was placed at the base of the sprue. There was also a control sample, in which no nodularization treatment was done. Sample designations are as shown in Table 6. Casting into the moulds was done at 1390°C, prior to pouring into the moulds, the chemical composition of each melt was determined for Mg content. The samples were allowed to cool after casting and finally finished. The chemical composition of the TWDI samples prior to casting was determined via spectrometric analysis using the spectrometer at the Foundry Laboratory of Nigeria Machine Tools Limited, Osogbo, Osun State, Nigeria.

Table 5: Summary of charge materials

S/N	Material	Quantity (Kg)
1	Steel Scraps	400
2	Ductile Iron Returns	3.8
3	Graphite Coke	89
4	Ferrosilicon (FeSi) alloy	7.2
	TOTAL	500

Table 6: Sample designations with wt. % Master alloy proportions

S/N	Sample Designation	Wt. % Master alloy
1	MA0	0
2	MA1	1
3	MA2	2
4	MA3	3
5	MA4	4
6	MA5	5

2.3 Microstructural Examination

The 3 mm cast samples that were treated with varying proportions of the Mg master alloys, were cut, ground and polished according to specifications outlined in ASTM E3 for metallographic analysis. The sequence of emery papers adopted for grinding were 240, 320, 400 and 600 grits, for polishing, an aqueous suspension of 0.05 micron alumina was adopted. They were then viewed using an optical metallurgical microscope in their unetched and etched conditions, the etchant used was 2% Nital solution. Nodularity (Equation 1) and nodule count was estimated using specifications outlined in ASTM A247 and E407 standard procedures.

$$\text{Nodularity} = \frac{\text{Number (area) of accepted graphite particles}}{\text{Number (area) of all graphite particles}} \times 100 \quad (1)$$

Nodule count (graphite nodules/mm²) is the quantity of nodules per square millimetres on a polished surface examined at X100 magnification.

2.4 Hardness Test

Brinell hardness test is carried out using a 10mm hardened steel ball indenter with a load of 3000kgf on a Foundrax/B.H.D/1003402 tester model located at Machine Tools Osogbo, Osun State, in accordance with ASTM E10 standard.

2.5 Tensile Test

Tensile property test was carried out according to ASTM E8 standard specification for flat samples, using a Universal Instron 3369 Tensometer, system identification number: 3369K1781, located at the Materials and Metallurgical Engineering laboratory, University of Lagos. The width of the samples was 6 mm, the thickness was 3 mm and gauge length was 30 mm. The image of the tensile test piece before fracture is shown in Figure 1. The test piece was loaded into the grips of the tensometer, the load was applied till the test piece fractured as shown in Figure 2. The force (F) applied and the elongation (ΔL) of the test piece are measured throughout the procedure.



Figure 1: Tensile test piece before fracture

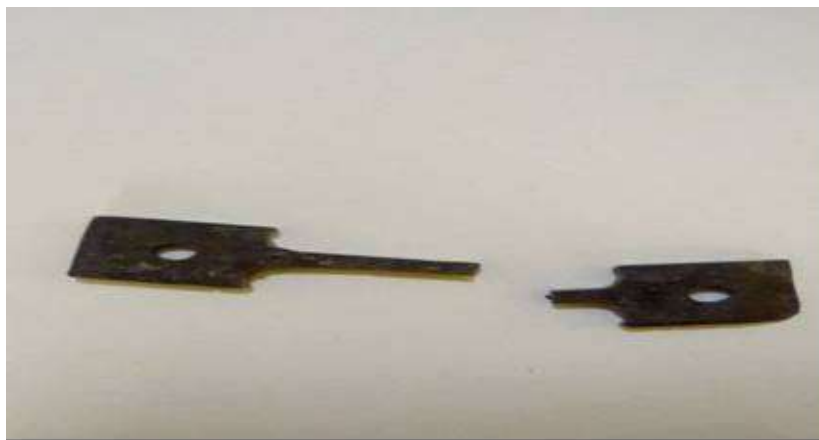


Figure 2: Tensile test piece after fracture

3. RESULTS AND DISCUSSION

3.1 Chemical composition of TWDI melt and Castings

Chemical composition of TWDI melt is shown in Table 7, from the Table, the chemical composition of Mg in the melt have been greatly impacted by the treatment using various master alloy proportions. It is important to note at this juncture that the iron melt treatment parameters and processing procedure were the same except for the proportions of master alloy employed for the treatment process.

Table 7: Chemical Composition of samples prior to casting

Sample Designation	Mg	C	Si	Mn	P	S	Fe
MA0	0.002	3.64	1.28	0.202	0.015	0.0021	94.85
MA1	0.027	3.65	2.28	0.208	0.012	0.002	93.82
MA2	0.033	3.69	2.45	0.211	0.011	0.002	93.60
MA3	0.039	3.58	2.79	0.147	0.008	0.0017	93.43
MA4	0.043	3.62	2.88	0.124	0.005	0.0011	93.32
MA5	0.047	3.67	3.13	0.119	0.003	0.0009	93.03

Proper melt treatment requires that residual magnesium (Mg) should be at least 0.03 wt. % to produce adequate nodule characteristics when high purity iron, carbon and silicon are used to produce the base cast iron. This implies that at least residual magnesium of 0.03% must be present in the casting as at the time it finally solidifies, so that it can sustain optimum nodule formation in the casting [10]. This implies that samples cast from MA1 and MA0 (control sample with no magnesium treatment) melts will not be capable to sustain adequate nodule characteristics. This tendency will impact negatively on nodularity and nodule count and consequently on the mechanical properties of the samples cast using these TWDI melt. The

melts for casting samples MA2, MA3, MA4 and MA5 on the other hand, have the capacity to sustain adequate nodule characteristics since the amount of residual magnesium is higher than 0.03 wt. %, i.e. adopting the amount stipulated for DI castings by [10]. For sample MA0, no nodularization treatment was carried out. This study seeks to establish the required proportion of a specified magnesium master alloy for casting TWDI sections where the sandwich treatment process is adopted. Investigation into the microstructural and mechanical properties of these cast samples should give better insight as to the impact of the various Mg master alloy proportions.

3.2 Microstructural Analysis

Microstructure of cast samples consisted of graphite nodules embedded in ferrite and pearlite matrix of varying proportions for samples that were treated with Mg master alloy whereas sample MA0 (Figure 3) showed graphite of flake like morphology. The micrographs of the samples are shown in Figures 3-8 for both the unetched and etched conditions. The micrograph for MA1 sample showed non-nodular and flake graphite precipitates, which could have been formed resulting from insufficient nodularization of nucleated graphite from the inoculation treatment. This shows that the proportion of master alloy employed for treatment (1 wt. % Mg master alloy) was not adequate to produce optimum graphite nodules hence the formation of some proportions of flake graphite structures. (Figure 4). With the increase in the proportion of the master alloy used for treatment the nodularity and nodule counts increased, a confirmation that melt treatment was adequate. Since all other melt processing parameters remained constant, it is evident that this graphite shape characteristics resulted from the adequate proportions of the master alloy employed (Figures 5 and 6). Increased graphite segregation was observed for MA2 sample (Figure 5) than in MA3 sample (Figure 6), this is responsible for larger volume of the ferrite phase in MA2 sample as observed also by the researchers in [11], and the nodule size was larger in MA3 than in MA2. This should impact on the hardness and percent elongation properties of MA2 sample. On further increase of the master alloy proportion to 4 and 5 wt. %, pyro effects increased and since magnesium is a strong carbide former, carbide precipitations in the microstructure was observed, also a decline in nodularity and nodule count occurred. These were observed more in the MA4 and MA5 samples (Figures 7 and 8). This trend was also the case in [6] where the researchers observed that with increased wt. % of Mg in the cast DI samples non-nodular i.e. spiky nodules were observed in the microstructure. Increased residual magnesium which resulted from an increase in Mg master alloy proportion did not result in better nodule characteristics but the formation of carbide precipitates which can negatively impact on mechanical properties of cast parts. These samples showed large proportions of carbide precipitations, shrinkages and non-nodular graphite in their microstructures. This trend was also observed in MA1 sample but to a lesser degree, attributable to inadequate magnesium treatment from small proportion of 1 wt. % Mg master alloy addition (Figure 4).

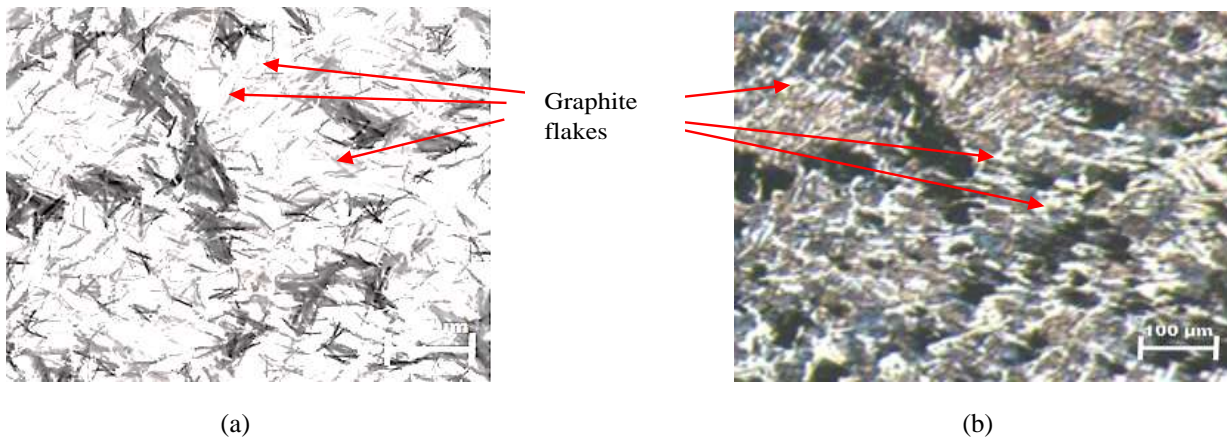


Figure 3: Optical micrograph of MA0 sample (a) unetched (b) etched

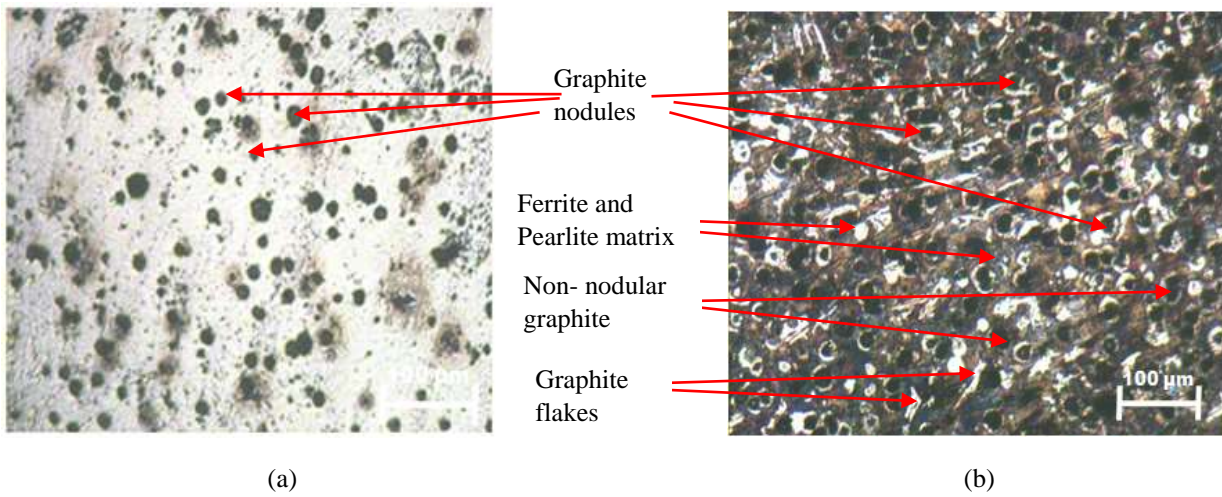


Figure 4: Optical micrograph of MA1 sample (a) unetched (b) etched

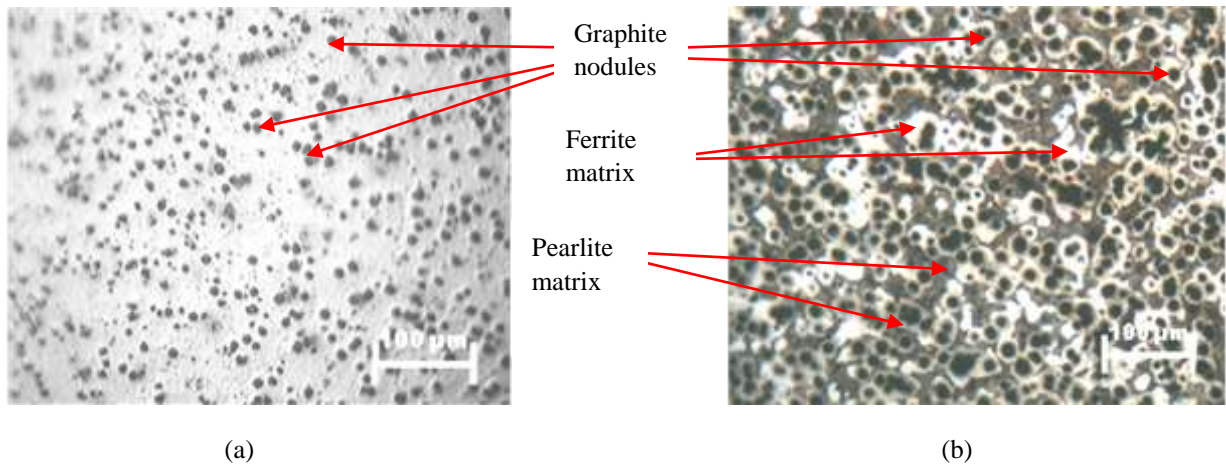


Figure 5: Optical micrograph of MA2 sample (a) unetched (b) etched

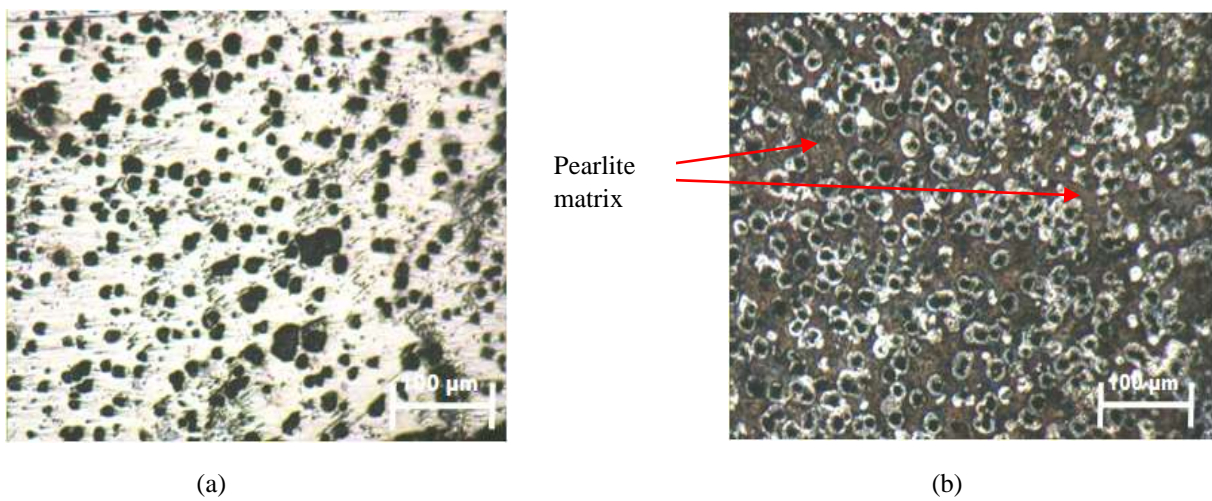


Figure 6: Optical micrograph of MA3 sample (a) unetched (b) etched

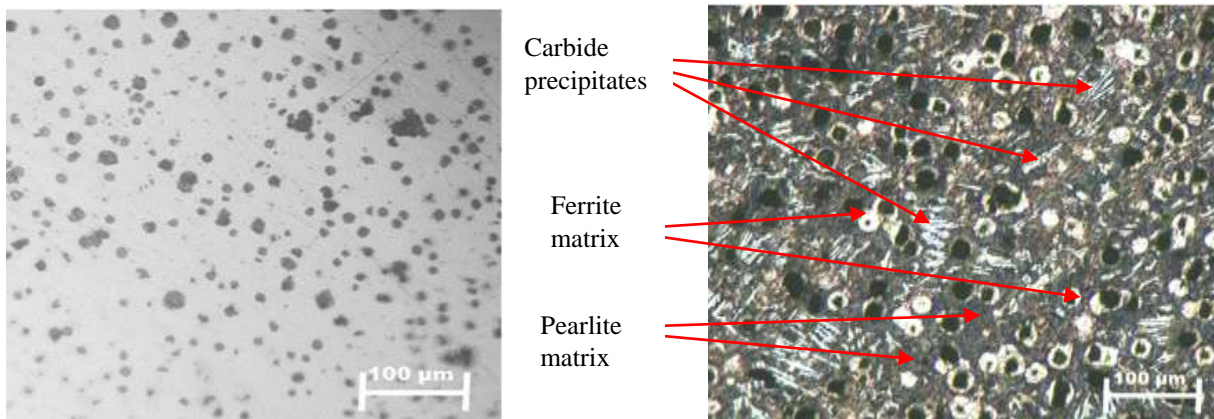
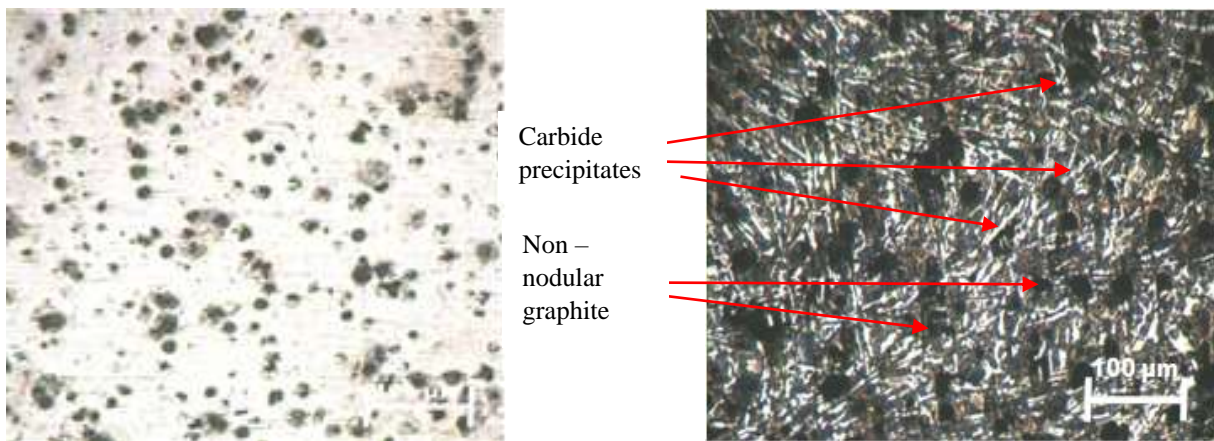


Figure 7: Optical micrograph of MA4 sample (a) unetched (b) etched



(a) (b)
Figure 8: Optical micrograph of MA5 sample (a) unetched (b) etched

3.3 Nodularity and Nodule Count

The nodularity and nodule count values were determined using manual procedure as outlined in ASTM A247 and E407 standard, these plots are presented in Figures 9 and 10. The increase in proportion of Mg master alloy impacted positively on nodularity and nodule count up for 2 and 3 wt. % Mg master alloy additions, giving 94.1%, 821 Nodules/mm² and 92.6 %, 735 Nodules/mm² for MA2 and MA3 samples respectively. Decline of these values was observed for MA4 and MA5 samples where these values dropped abruptly to 61.3 %, 52.4 % and 324, 208 Nodules/mm² for nodularity and nodule count values respectively, similar trend was also observed by the study in [6]. For the MA0 sample, no nodules were formed, the structure had only flake graphite structures. This shows that 2, 3 wt. % master alloy additions produced adequate nodularity and nodule count ratings, so depending on the DI grade required, either of these proportions could be adopted. Defects such as carbide precipitations, shrinkages and non-nodular graphite precipitations are observed in the microstructure of MA4 and MA5 samples, these impacted negatively on nodularity and nodule count ratings. With increasing magnesium content, magnesium recovery in liquid iron decreases while pyro effect increases, so the lower master alloy proportions in MA2 and MA3 samples led to lesser pyro-effects, reduced oxidation, better magnesium recovery and also reduction in the carbide forming tendency, these factors contributed significantly in increasing the residual magnesium available for adequate nodule formation with better shape characteristics.

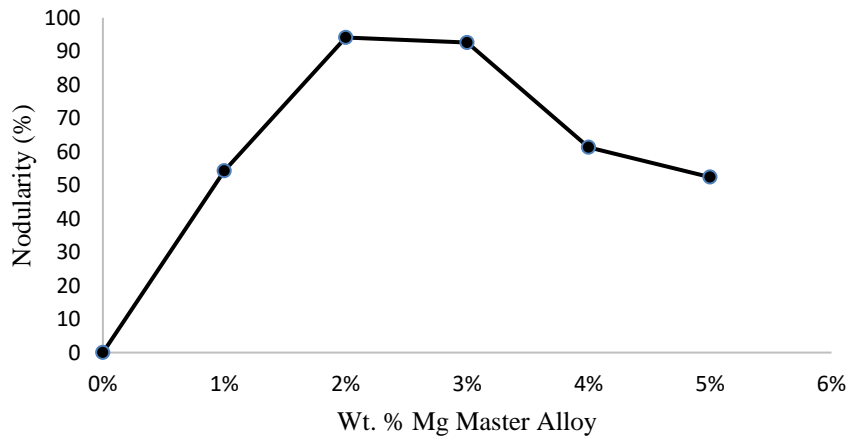


Figure 9: Variation of Nodularity of cast samples with proportion of Mg master alloy

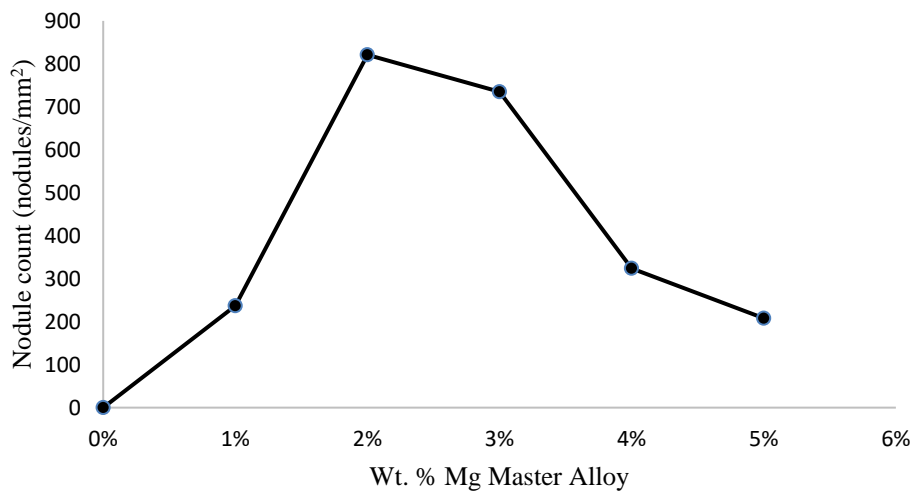


Figure 10: Variation of Nodule count of cast samples with proportion of Mg master alloy

3.4 Hardness Analysis

The variation of BHN with wt. % master alloy of cast TWDI samples is shown in Figure 11. The control sample which solidified with flake graphite structure recorded 187 BHN, then a downward trend in hardness was observed for 1 and 2 wt. % master alloy additions, attributed to the formation of graphite nodules/ spheroids, i.e. 199 BHN for MA2 sample. This value picked up again to 211 BHN with MA3 sample due to the increased pearlite proportion in the structure [12]. Also larger proportions of ferrite phase in MA2 which resulted from increased graphite segregation (higher nodularity and nodule count) gave rise to the slight reduction in hardness property of MA2 than MA3 sample i.e. from 199 BHN to 211 BHN for MA2 and MA3 respectively. Higher values were observed in MA4 and MA5 samples due to larger proportions of the harder carbide phases present as Mg is a strong carbide former and also increased pyro effect. Depending on the desired TWDI grade, the proportion of master alloy could be selected by the Foundrymen to produce ASTM A536 grades 80/50/06 and 100/70/03.

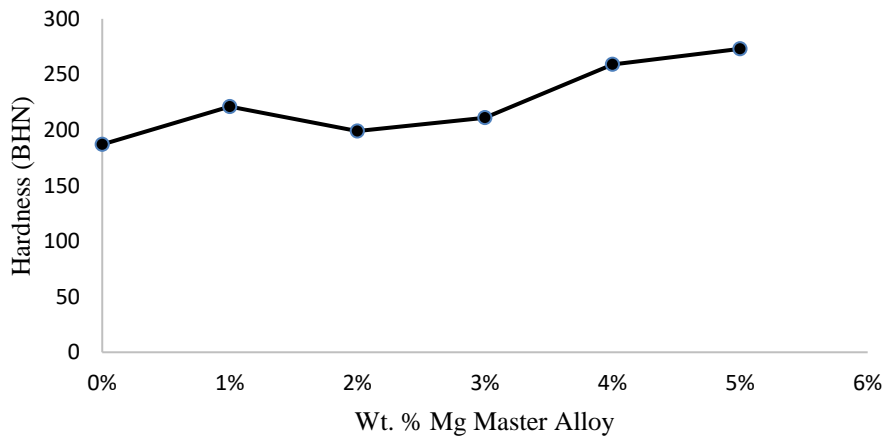


Figure 11: Variation of BHN of cast samples with proportion of Mg master alloy

3.5 Tensile Test

The plots for the tensile analysis of the samples with Mg master alloy proportions are shown in Figures 12 and 13 for ultimate tensile strengths (UTS) and percent elongation respectively. The microstructure of sample MA0 was that of graphite flakes in ferrite and pearlite matrix which is similar to that of grey cast iron recorded 314 MPa, this value increased with increase in percent Mg master alloy employed for both MA1, MA2 and MA3 samples, after which it dropped to 433 MPa for MA4, and then dropped further to 334 MPa for MA5. The increased volume of pearlite phase attributed to the rise in UTS from MA2 to MA3. The trend showed that increased Mg master alloy proportion produced a proportionate increase in UTS value up to 3 wt. % addition during the nodule treatment and afterwards initiated the formation of carbide precipitates which reduced the tensile strength of the samples. From the percent elongation plot in Figure 13, it was observed also to increase with increased master alloy adopted for the melt treatment to MA2, after which there occurred a decline from 6.1% for MA2 to 4.2% for MA3, and even further declined to 2.7% and 2.1% for MA4 and MA5 samples respectively. The percent elongation values had an inverse relationship with that of the BHN values.

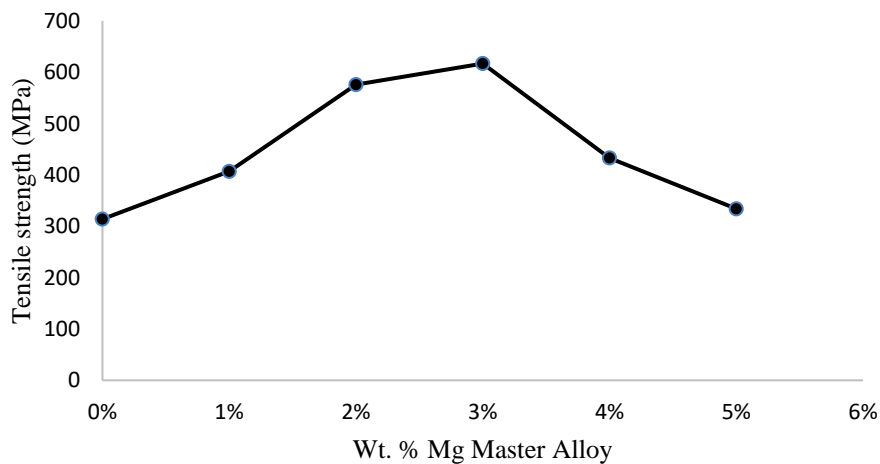


Figure 12: Variation of Tensile strength of cast samples with proportion of Mg master alloy

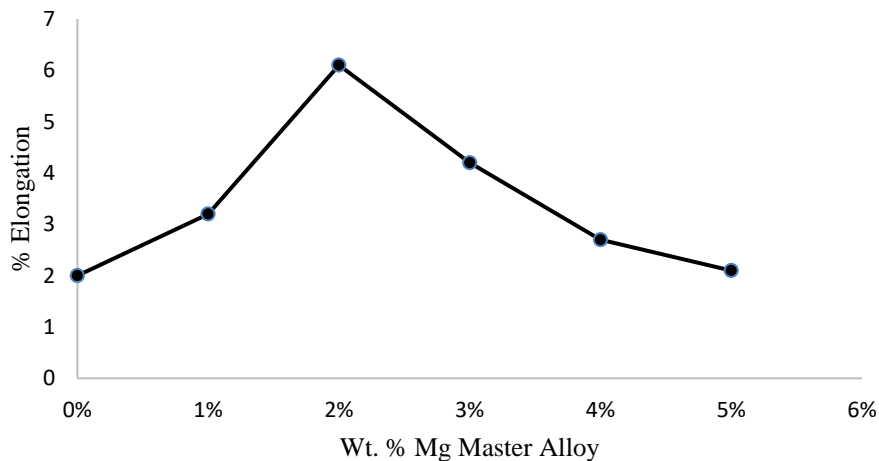


Figure 13: Variation of Percent elongation of cast samples with proportion of Mg master alloy

This study has shown that 2 and 3 wt. % Mg master alloy gave optimum microstructure and mechanical properties in accordance with the specifications outlined in ASTM A536 Ductile Iron grades, precisely grades 80-55-06 and 100-70-03. Many of our indigenous Foundries adopt the trial and error approach for the proportion of Mg master alloy to be used in the Sandwich melt treatment process as they lack reliable source of information to enable them select the right type and proportion of Mg master alloy. The standardization for this proportion cannot be overemphasized as this treatment is crucial in producing defect free DI castings, also for TWDI parts with their peculiar fast cooling rate [11], even more stringent proportions of Mg master alloy is germane in order to produce optimum nodule characteristics, mechanical properties and the desired grade. Also the use of Mg-master alloys of same nominal composition, but produced by different manufacturers will result in varying level of properties depending on Mg-treatment method employed i.e. sandwich or in-mould treatment processes. Foundrymen do not have reliable source of information allowing them to select the right Mg-master alloy depending on Mg treatment method used in the particular Foundry. [13]. This study which was aimed at proffering Mg master alloy proportions to be adopted in sandwich treatment based on the chemical composition of the Master alloy adopted has shown that 2 and 3 wt. % additions are appropriate. Based on the results obtained for nodularity and nodule count, matrix type present, hardness value, ultimate tensile strength (UTS) and percent elongation, the proportions to be adopted will range from 2 - 3 wt. % Mg master alloy. It is unlikely to achieve adequate Mg recovery at higher wt. % Mg master alloy due to increased pyro-effect tendency and carbide precipitation.

4. CONCLUSION

This study has shown that in adopting the sandwich treatment process and using the Mg master alloy with the composition given in Table 4, 2-3 wt. % master alloy is adequate in producing TWDI (3 mm) with microstructures and mechanical properties outlined in ASTM A536, DI grades 80-55-06 and 100-7-03. Matrix structures ranging from ferrite, pearlite and carbide precipitates of various proportions were obtained in the as-cast samples. High nodularity and nodule counts ratings were achieved for 2 and 3 wt. % Master alloys additions i.e. MA2 and MA3 samples. Control sample MA0 where no nodularization treatment was carried out had no graphite nodules instead graphite flakes were observed. However 1, 4 and 5 wt. %s showed carbide precipitates in their microstructure, with largest proportion of this phase in MA5 microstructure, which impacted negatively on their mechanical properties. This implies that using higher proportions of the master alloy from 4 and 5 wt. %s does not produce higher nodularity and nodule count ratings rather carbide precipitations occurred which can be attributed to the fact that Mg is a well-known carbide former/promoter in iron melts. Also it is observed that pyro-effect increased with adoption of higher proportions of the treatment alloy. This phenomena led to lower Mg recovered in the melt for optimum nodularity and nodule count ratings. High solidification rates experienced when TWDI parts are cast implies that strict and accurate melt processing parameters are adhered to, this study has therefore contributed in the area of specifying Mg treatment alloy proportions necessary for the casting of defect free TWDI parts.

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