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Original article

Structural characterization and mechanical properties of pearlite – Enhanced micro-alloyed ductile irons

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ABSTRACT

The structural characteristic and mechanical properties of ductile irons micro alloyed with lean additions of molybdenum, nickel, copper and chromium was investigated. This was aimed at assessing the potentials of the utilization of lean ferro alloy additions (which offers reduced processing and product costs) for enhancing pearlite phase proportion, which is required for improved mechanical performance of ductile irons. The ductile irons contained a maximum of 0.2% each of Mo, Ni, Cu, and Cr and were processed using a crucible furnace. They were characterized using optical microscopy and X-ray diffractometry while hardness and tensile testings were used to evaluate the mechanical properties. The results show that the micro alloyed samples contain new compound of alloying elements with iron and the base alloy phase (FeSi, α Fe). It was also observed that the micro alloy additions resulted in significant increase in pearlite proportion from 30.63% in the base alloy to as much as 59.38% in the composition containing Mo, Ni and Cu as micro addition. Increase in hardness within the range 1.4–36.5% was obtained, while tensile strength increase within the range 35.89–80.55% with the use of the micro alloying additions. Overall, the best combination of mechanical properties was achieved for the ductile irons composition containing chromium and copper, as well as the one containing molybdenum, nickel and chromium as micro alloy additions.

1. Introduction

Ductile iron is a class of cast iron characterized by the existence of graphite in nodules which are dispersed in the iron matrix. The nodular graphite morphology imparts great improvement in mechanical properties in ductile irons, which is preferred in automotive industry than other class of cast iron such as compacted graphite iron as a result of tis improved mechanical properties (Soliman et al., 2015; Hou et al., 2017; Fragassa et al., 2016; Fragassa and Pavlovic, 2016). Therefore there is the need to obtain the required matrix that will be able to give adequate strength. Depending on the specific application, the ductile iron matrix structure can be tailored to be martensitic, pearlitic, ferritic or a combination of pearlite and ferrite, this is possible through the application of heat treatment and alloying (Kiani-Rashid, 2009a; Gonzaga, 2013). According to Gonzaga et al. (2009) and Dicocco et al. (2010) alloying has the advantage of stabilizing the desired phases making it possible to preserve microstructures (matrix structure) of choice. Mohammed (2016) however reported that the use of alloying can be associated with an increased tendency for elemental segregation and undesired carbide formation in ductile irons which alters the overall expectations in terms of engineering properties. In order to reduce this tendency of carbidic phase formation, the use of micro alloying addition not exceeding 0.5% have been explored in the development of the ductile irons (Mohammed, 2016). Rao et al. (2014) investigated the influence of composition ratio of manganese and copper on the mechanical properties and machine performance of ductile iron. It was reported that both manganese and copper promote pearlite formation in ductile iron with increased tensile strength and average hardness. However, the strengthening ability of copper in ductile iron is noted in their findings to be effective when the manganese content of the ductile iron is low. Mohammed (2016) also investigated the influence of manganese, nickel, molybdenum and copper

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Table 1
Chemical Compositions of the Melts Produced.

Melt	CE	% C	% Si	% Mn	% Mo	% Ni	% Cr	% Cu	% Mg	% S	% P
A	4.27	3.42	2.50	0.36	0.11	0.16	–	0.20	0.073	0.032	0.048
B	4.38	3.50	2.60	0.39	0.19	0.22	–	–	0.086	0.034	0.042
C	4.14	3.20	2.80	0.53	–	–	0.12	0.21	0.095	0.026	0.031
D	4.23	3.40	2.45	0.50	0.24	0.18	0.11	–	0.091	0.031	0.048
E	4.18	3.30	2.62	0.42	–	–	–	–	0.081	0.027	0.029

above 0.5% on the mechanical properties of ductile irons, and reported that high hardness and strength could be achieved but there was formation of carbides on the grain boundaries, which will impair the achieved properties in service at higher loads. Gonzaga (2013) investigated the influence of pearlite and ferrite on mechanical properties of ductile irons and concluded that possession of pearlite matrix phase in appreciable amount in iron helps to enhance the mechanical properties. Based on the above premises, it is observed that the alloying combination greatly influences the ductile iron matrix structure. This necessitated the use of lean additions of alloying elements at not more than 0.5% (micro alloying) for enhancement of mechanical properties in ductile iron, as this has not been widely reported. Therefore the present study is premised on enhancing pearlite volume fraction as a basis of improving the mechanical properties of ductile irons. The investigation assesses the influence of micro alloying elements combination (Mo, Cu, Cr, and Ni) on the matrix structure and nodule count, and how this affects the mechanical properties of the ductile irons. The result of this investigation is instructive in determining the required combination of the micro alloying additions needed to achieve significantly improved mechanical properties in ductile irons.

2. Materials and methods

2.1. Materials

Materials used for this research work are: automobile gray cast iron scraps, graphite, ferrosilicon magnesium (5% Mg, 45% Si) ferromanganese (80% Mn), ferrochrome (64% Cr) nickel metal, ferromolybdenum (72% Mo) copper (copper wire 98%), flux (calcium carbonate) and calcium carbide.

2.2. Ductile irons production

Melting of grey cast iron scrap was carried out in an oil fired lift out crucible furnace. The charges were prepared with mixed proportions of alloying elements for each sample composition. This was done following standard procedures in accordance with (Khanna, 2009; Soiński and Góraj, 2009). The charged materials were heated in a graphite crucible to temperature of 1300 °C before addition of calcium carbide and stirred to desulphurise the melt. The melt was superheated to temperature of 1420 °C, treated with magnesium ferrosilicon (5% Mg, 42% FeSi) in a ladle according to sandwich process and cast in green sand mould after treatment at the stated temperature of 1420 °C, which is in accordance with Ramadan and Fathy (2014). The cast samples were cylindrical rods with dimension of Ø 20 mm × 200 mm long.

2.3. Mechanical testing

2.3.1. Hardness measurement

Hardness measurement was done on prepared samples using INNOVATEST FALCON 500 micro hardness testing machine in accordance with ASTM E29 – 16 standard. Test load of 0.1 Kgf was applied on each sample with dwell time of 10 s. Five hardness indents were made on each sample and the reading within the margin of ± 5% were taken for calculating the average of the five readings as the hardness value of each ductile iron composition, which is also in accordance with (Shayesteh-Zeraati et al, 2010).

2.3.2. Tensile testing

Tensile properties were evaluated using MTS STH tension meter in accordance with ASTM E8M – 15 standard. Specimens for the test were machined to tensile specification of 12.5 mm diameter and gauge length of 50 mm. The specimens were mounted on the test platform and pulled at a strain rate of 10^{-3}/s .The test was carried out three times on each ductile iron composition and the average result of the three experimental tests were used. The tensile properties evaluated from the test are the ultimate tensile strength, strain to fracture and percentage elongation.

2.3.3. Chemical composition determination

The chemical composition of the ductile irons produced was determined using Tasman absorption spectrometer with argon gas accessory, which operates by spark action on the surface of the specimen to be analyzed. Specimens surface were first prepared by grinding and obtaining flat surface. Average of three readings with variability of at most ± 7% value, was taken as the composition of the specimen measured. Result of composition measurement is presented in Table 1.

2.4. Structural characterization

2.4.1. X-Ray diffraction

PANanalytical XRD machine with empyrean diffractometer equipped with PIXel detector at fixed slit was used for the analysis. The slit was fixed with Fe filtered Co-Kα radiation with a rotating head anode which was used to scan the angular 2θ range of 0 to 90^{O}.The phases were identified using X'pert highscore plus software.

2.4.2. Optical microscopy

Zeiss optical microscope with Axiocam5 camera attachment was used for microstructural analysis of the ductile iron produced. Specimens for microstructural analysis were prepared metallographically through the process of grinding and polishing.Thereafter, the specimens were etched in 4% nital , swabbing for 10 to 15 s after which the microstructures were examined using the microscope. The phases present in the microstructures were quantitatively analyzed using ImageJ software application.

3. Results and discussion

3.1. 3.1 chemical composition analysis

The chemical composition of the ductile irons produced is presented in Table 1. It is observed that the silicon content in the melt were within the range 2.45 – 2.80%, which is adequate to prevent melt chill, and also sufficient to facilitate production of enough nodules in the melt and prevent segregation (Kiani-Rashid, 2009b; Ochulor, et al., 2010). Residual magnesium in the ductile irons produced is within 0.073 – 0.095% which is more than the minimum residual magnesium of 0.05% that can sustain the production of nodules in a melt (Tiedje, 2010). There is high residual magnesium left which was not consumed in the course of nodulization and desulphurization reaction during treatment. It then indicated that nodules formation was going on as part of the magnesium was also undergoing reaction with sulphur to form MgS in the slag phase (Akinlabi and Omole, 2014). The carbon equivalent values of the ductile irons is within the range of 4.14–4.38, which is close to the eutectic composition and thus enhance good fluidity of the melt (Brown, 1994).

3.2. X-ray diffraction

The X-ray diffraction patterns of the ductile irons produced are presented in Fig. 1. The results show the presence α-Fe phase with crystal lattice plane (1 1 0), in addition to several Fe based compounds. FeSi and $Fe_{19}Mn$ were dominant phases observed in all the ductile irons with the FeSi diffracted alone crystal lattice planes of (2 0 0) and (2 1 1). $Fe_{9.7}Mo_{63}$, Cu_3Fe_{17}, Fe-Cr, $Fe_{19}Mn$, FeNi are some of the other phases observed in the XRD patterns of the ductile irons produced.

3.3. Microstructure analysis

Micrographs of the ductile irons produced and the results of image (phase) analysis are presented in Fig. 2 and Table 2, respectively. It is observed that all the ductile irons produced contained predominantly pearlite as matrix phase. However, the amount (proportion) of pearlite increases with the presence of micro alloying addition (0.15% of Mo, Ni, Cu and Cr). The results indicated that addition of micro alloying elements have influence on the resulting microstructure obtained (Kawalec and Kozana, 2014). The pearlite formed in the ductile irons containing alloying elements increases within the range of 62.3 to 93.9% in comparison with theone without alloying elements. This increase was due to the presence of the micro alloying elements which undergone complex reactions with each other within the matrix, which later transforms into tiny pearlite particles that are distributed in the casting (Rao et al., 2014). Nodules were contained in the structures of the irons, which is an indication of the effectiveness of the sandwich process utilized in adding magnesium into the melt (Riposan et al., 2003). The nodules are observed to be well dispersed in the ductile iron matrix. The nodules count and nodularity of the structures developed are good indicators of good mechanical properties in ductile irons (Ochulor et al., 2015).

3.4. Mechanical properties

3.4.1. Hardness

It is observed from Fig. 3 that all the ductile iron compositions with micro alloying additions have hardness values greater than that of the base ductile iron composition (340 HV/0.1). The hardness increase relative to that of the base composition ranged between 1.4 and 36.5%, with the composition containing Mo, Ni and Cr having the highest hardness value. The effect of various alloying elements added was noticed in the increase in pearlite formation in the micro alloyed iron as compared to control samples without alloying elements. For instance samples C and D have pearlite values of 56.06% and 56.59% respectively, as against 30.63% in base metal (Table 2). They contained the highest pearlite and relatively the smallest ferrite contents; hence they have the highest hardness of all the samples. The improved hardness in the micro alloyed ductile iron compositions can be attributed to lower ferrite phase content in these compositions (29.83 – 40.11%) compared with the base ductile iron composition which consist of 59.37% ferrite. The ferritic structure is known to be a soft phase, so it is logical that the composition with the highest ferrite content exhibits the least resistance to indentation (lowest hard-

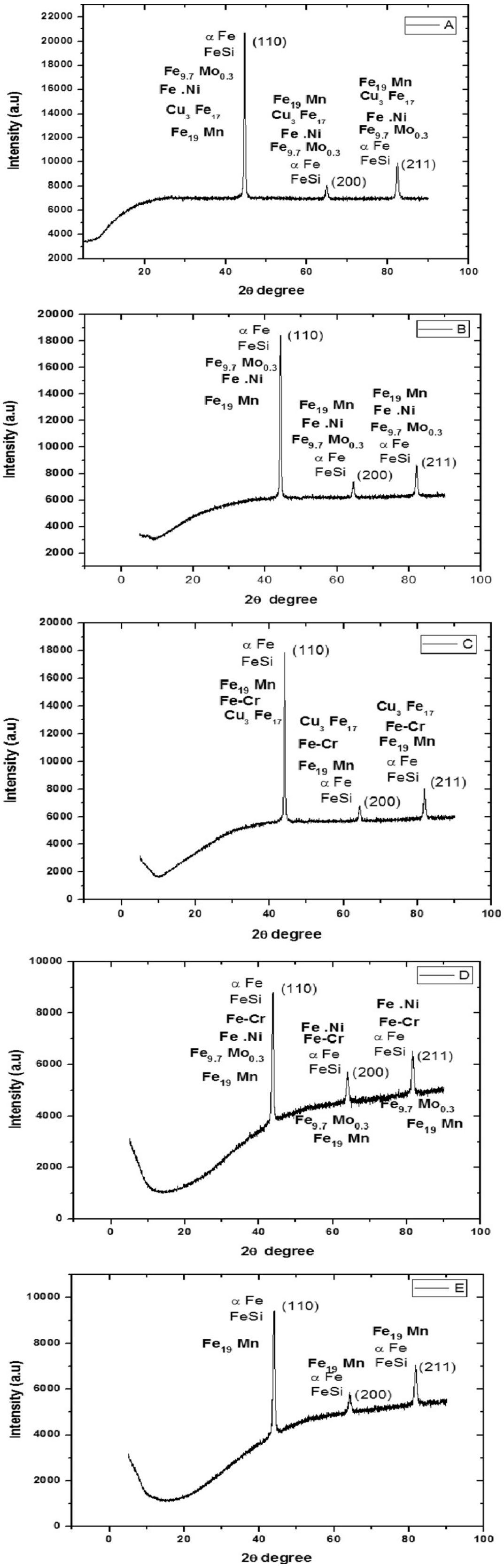
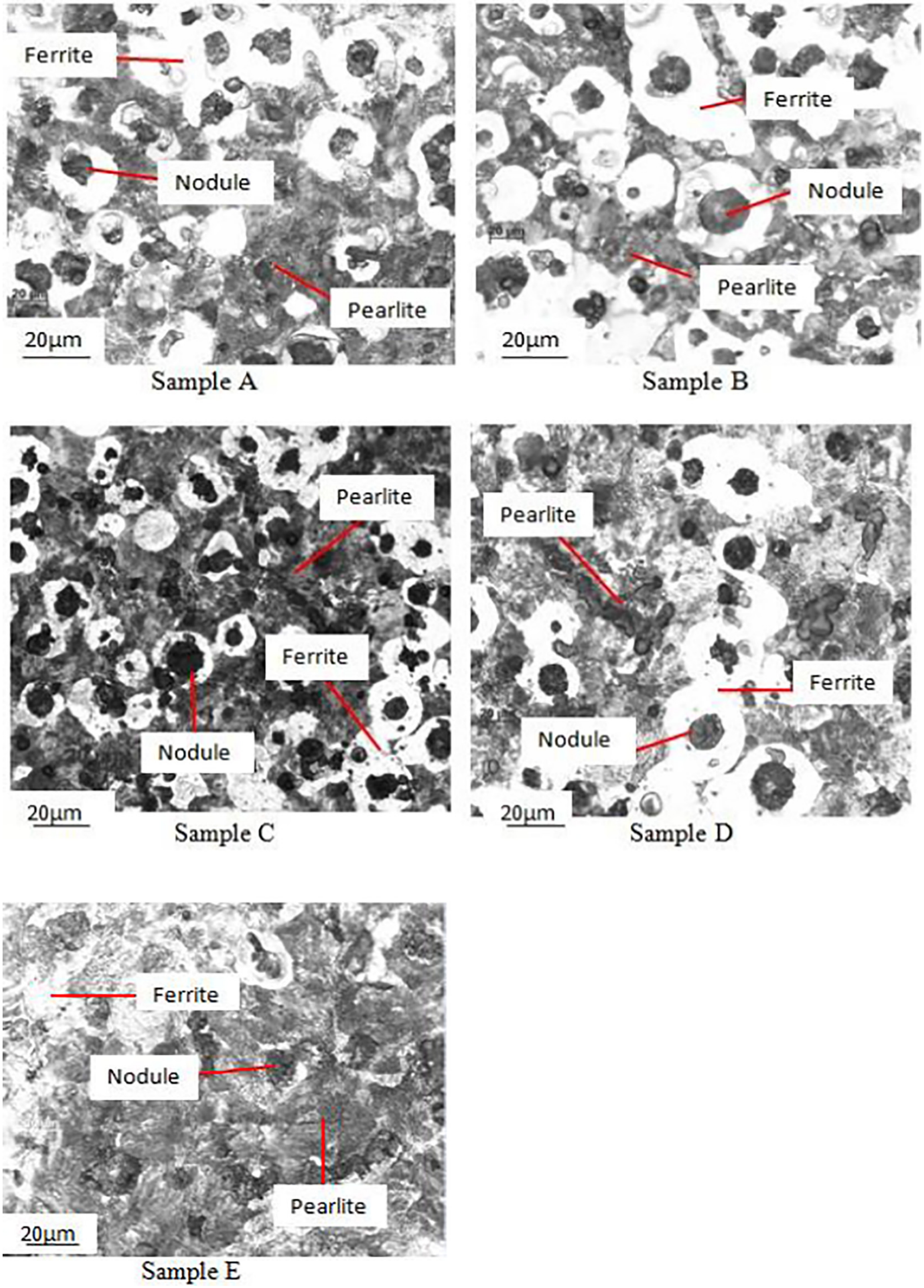


Fig. 1. The XRD Pattern of all the Ductile Irons Produced.



Sample A

Sample B

Sample C

Sample D

Sample E

Fig. 2. Optical Micrographs of the Ductile Irons Produced.

Table 2
Microstructural Analysis of the Phases and Nodules Area Fractions of the Ductile Iron.

Sample	Volume fraction of Pearlite	Volume fraction of Ferrite	Volume fraction of Nodule	Nodularity %	Nodules count (per mm^2)
A	49.38% ± 2.55	38.93% ± 2.35	11.14% ± 1.82	91	110
B	49.70% ± 2.82	40.11% ± 2.64	10.98%±1.68	90	115
C	56.06% ± 1.85	29.83% ± 2.43	14.51%±2.08	88	105
D	56.59% ± 2.32	32.68% ± 2.42	11.31%±1.95	92	120
E	30.63% ± 2.12	59.37% ± 2.71	10.27% ± 2.10	88	107

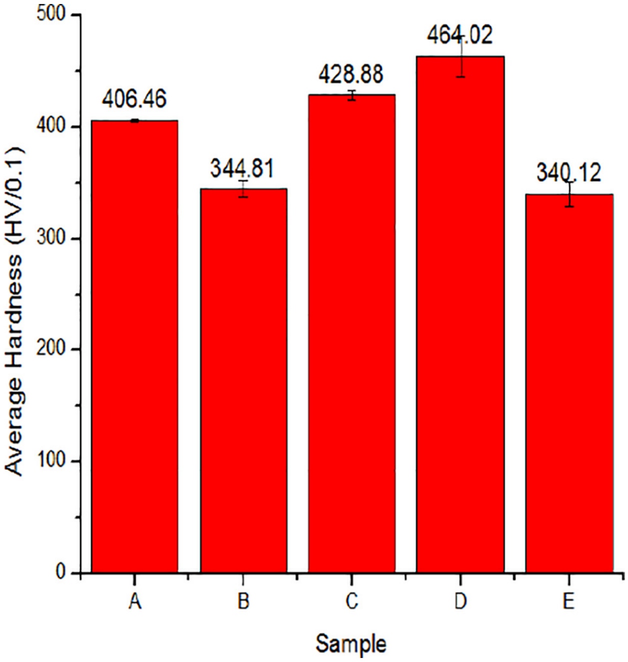


Fig. 3. Variation of Average Hardness of Ductile Irons Produced.

ness). Also, increase in pearlite phase is synonymous to increase in hardness value. This is because pearlite phase is a relatively hard phase which is utilized to increase the hardness and mechanical properties in general.

3.4.2. Stress – strain

The stress – strain profiles of the ductile irons produced are presented in Fig. 4. It is observed that samples C and D show higher work hardening response compared to other ductile irons composition produced. The ultimate tensile strength and percent elongation as extracted from the stress-strain profile are presented in Figs. 5 and 6, which show more precisely the tensile behaviour of the ductile irons produced.

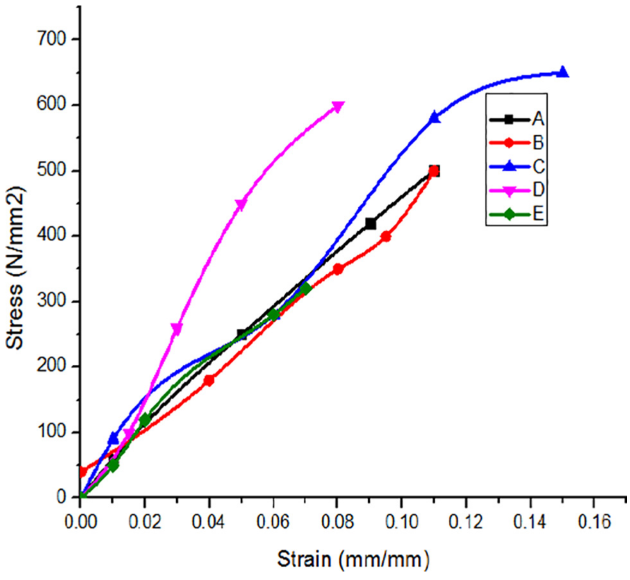


Fig. 4. Stress – Strain curves of all the specimens.

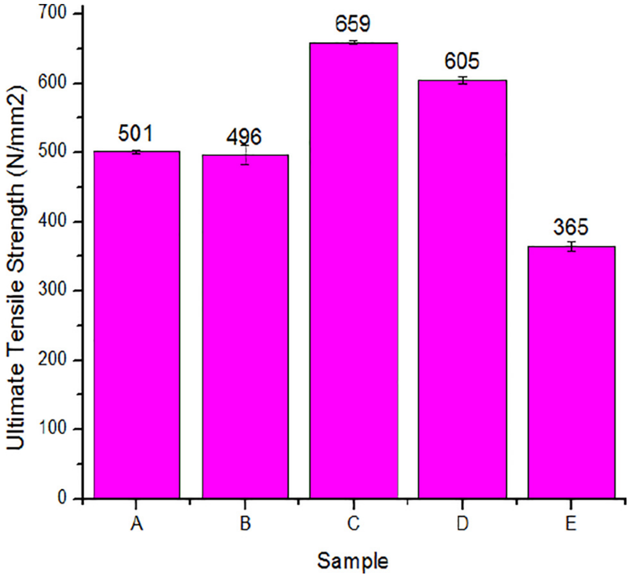


Fig. 5. Variation of Ultimate Tensile Strength.

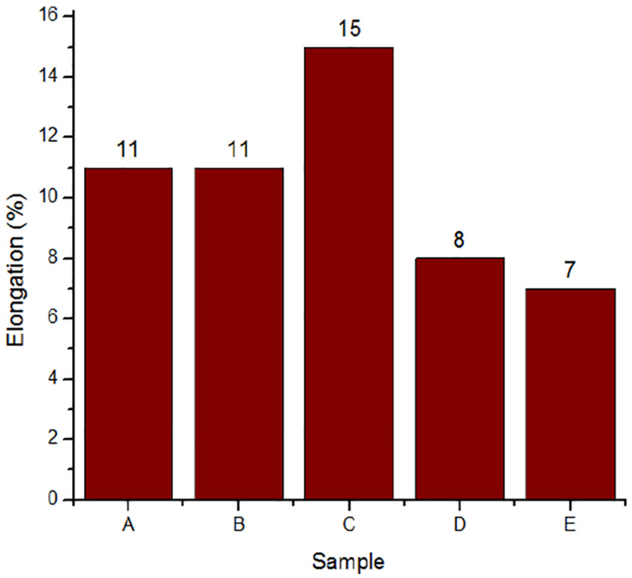


Fig. 6. Variation of Percentage Elongations of the Ductile Irons Produced.

3.4.3. Ultimate tensile strength

Variation of the ultimate tensile strength of the ductile irons produced is presented in Fig. 5. It is observed that there is an increase in strength of ductile irons containing micro alloying elements from within 35.89% to 80.55% as compared to ductile iron without alloying elements. This can be attributed to the synergistic effect of high nodules count, high pearlite phase content and relatively low ferrite phase relative to the base ductile iron composition without alloying elements. For instance, increase in volume fraction of nodules in samples C and D in comparison to that without alloying elements are 41.3% and 10.12% respectively. These outlined factors made sample C which contained Cr and Cu and sample D which contained Mo, Ni and Cr possess superior mechanical properties. The combined effects of chromium and Copper in sample C and that of molybdenum, nickel and chromium in sample D, facilitated the increased pearlite content observed above other ductile iron compositions, and consequently resulted in improved strength of the ductile iron.

3.4.4. Percentage elongation

The percentage elongation values of the ductile irons produced are presented in Fig. 6. It is observed that the control sample (composition without alloying elements) has relatively lower percent elongation compared to other ductile iron compositions containing alloying elements. This observation appears to negate conventional expectation, considering the fact that the control sample also had the lowest ultimate tensile strength and hardness. The input deducible from the observation is that despite the increased matrix strengthening achieved from the use of the lean micro alloying additions, they did not result in the development of embrittling phases, which could hamper the ductility of the ductile irons. The results show that they rather helped in achieving an improved combination of strength and ductility in the ductile irons.

4. Conclusion

Micro alloying addition with chromium, molybdenum, copper and nickel with manganese in base metal was carried out on ductile iron. The resulting microstructures, characterization and mechanical properties- hardness and tensile was investigated. The results show that:

1. The volume of the pearlite structure contained in each casting increased in the samples that were alloyed as compared to the control sample that contained no major alloying elements used. This is because alloying elements such as molybdenum, manganese and copper are pearlite promoter when used in regulated amount in other not to encourage the formation of stable carbide. In addition copper helps to suppress carbide formation.
2. The hardness value, ultimate tensile strength of the ductile irons increased significantly in alloyed samples containing chromium and copper addition than others. This is due to the combined action of the two elements which did not only help in formation of pearlite matrix but strengthened the irons. Small amount of chromium improves tensile strength and hardness and also stabilizes pearlite formation. Invariably, formation of appreciable pearlite in casting by this alloying system helps to strengthen as well as increase the hardness of the product.
3. X-ray diffraction results show formation of compound of the alloying elements with iron, in addition to the compound formed in base iron. It therefore show the inclusion of each alloying elements and their combination with the iron (Fe) in the various phases and peaks formed.

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