



# Multiple inoculations of ductile iron and the effects on properties

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## Abstract

Multiple inoculation of ductile iron and the effects on the structure and mechanical properties have being investigated. Samples of ductile iron were produced with inoculation carried out either once or twice and with different materials as inoculants. Ferrosilicon was used for the primary inoculation and either ferrosilicon or nickel-ferrosilicon for the secondary inoculation. It is observed that the nodules produced are more and finer with multiple inoculations and the effect is more pronounced with nickel-ferrosilicon as the secondary inoculant. Multiple inoculations produce an increase in the hardness of ductile iron when ferrosilicon is used as the secondary inoculant while a decrease in the hardness is observed with nickel-ferrosilicon despite the finer nodules. This is explained by the fact that nickel enhances graphitization in cast iron thereby depleting carbon in the matrix and making the cast iron weaker but with more nodules.

**Keywords:** Multiple Inoculations; Nodular Iron; Inoculants; Microstructure; Mechanical Properties.

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## 1. Introduction

Ductile Iron, a member of Cast Iron family, since its discovery has been found to be very dynamic by its lower temperature castability than steel and improved derivable mechanical properties when heat-treated [1, 2]. It is an alloy of Iron-Carbon-Silicon with some alloying elements (Mn, Ca, Ni, Mo, Cr, etc.) added as a means of controlling the matrix microstructure [3]. Therefore, it is the carbon and silicon that determine largely the microstructure of the as-cast product [4]. Carbon exists either as graphite or cementite in cast iron and the form it exists is a function of the silicon content and cooling rate during solidification [4, 5]. Silicon composition above 2% is prescribed for ductile irons. The silicon content is judged to be the most important determinant of matrix carbon content [3]. It is a graphitizer and causes the instability of cementite [6] by decomposing it to yield graphite [4]. Another important graphitizer in cast iron is nickel and graphite is a very soft and weak material [5].

The morphology of the graphite in cast iron varies. It can be flake-like (as in grey cast iron), nodular (as in ductile cast iron) and exploded [7]. The graphite flake in grey cast iron causes relatively low strength, with its edges serving as stress raisers and almost non-existent ductility [4, 7]. Ductile iron, chemically the same as grey iron and developed to overcome the brittle nature of grey iron, is an improvement on gray cast iron. It is also known as nodular or spheroidal iron because it has its graphite in nodular form or as distinct spheroids whose morphology serves as crack arrester since cracks have no convenient weak path through which to propagate [8]. Consequently, it is prone to have relatively higher mechanical properties than gray cast iron [2, 8, 9]. Further improvement on properties can be brought about by processing and heat treatment to obtain a wide range and superior combination of mechanical properties [1, 4, 10, 11, 12, 13].

Ductile iron can be produced from grey cast iron by adding small quantity of a nodulariser to molten grey iron under controlled conditions just before casting [1, 4]. Nodularisers change the flake-like to nodular morphology. Magnesium is the most popular foundry nodulariser but it is vigorous and violent in reaction with molten iron [3, 4, 9, 14]. It is

added as a master alloy (like MgFeSi) containing about 5% magnesium to reduce the violence of the reaction [6, 9]. About 0.03 to 0.05 weight percent of magnesium in the cast iron is found to be optimum for successful nodularisation [4]. However, some researchers have reported the production of ductile iron using calcium, calcium carbide, calcium fluoride, lithium, etc [1, 4, 15]. Imasogie, et.al. [1, 15] have produced ductile iron using master alloys containing calcium and magnesium, Ca-CaC<sub>2</sub>-Mg and CaSi-CaF<sub>2</sub>, with success. Nodularisation involves the removal of some surface active elements like sulphur, phosphorus and oxygen [4]; hence, nodular iron should have less sulphur and phosphorus than grey iron. Successful nodularization should yield a very good effect in the production of ductile iron of acceptable nodularity, usually between 85% and 100% [9].

Inoculation process is an integral part of ductile iron production [14, 16]. Practically, inoculation involves the use of certain materials that, when added to molten iron prior to casting, makes higher quality, more predictable ductile iron castings [16]. Nodularisation will be of benefit only when there are nucleation sites for the graphite nodules to nucleate and grow. Inoculation facilitates the formation of these nucleation sites, prevents carbide formation and precipitates graphite as nodules [16, 17]. The grains of graphite nodules are separated by grain boundaries. Inoculation increases the number of graphite nodules, assists in the formation of ferrite and promotes ductility [4].

Inoculation is carried out at various stages of ductile iron production. The practice is to add the inoculant when the ladle is 25 to 75 percent full, for primary inoculation. Further inoculation intensifies the primary inoculation effect and it is done at the point of casting [4]. It generates finer grain size and consequently higher strength, according to the relationship in Equation (1).

$$\sigma_y = \sigma_o + kd^{-1/2} \quad (1)$$

Where  $\sigma_y$  is the yield strength,  $\sigma_o$  and  $k$  are material constants and  $d$  is the average grain diameter. Moreover, grain refinement is the most effective means of improving both strength and toughness [18].

The amount of inoculant added varies from 0.2% for the majority of grey irons to 0.75% for the critical irons [4]. The most popular inoculant is ferrosilicon with the best effect being when it is used with some minor elements [14]. Conventional inoculants based on ferrosilicon alloys containing additives like calcium, magnesium, barium, zirconium, bismuth etc have been employed [14, 17]. However, a new inoculant concept, Ca, Ce, S, O, bearing ferrosilicon inoculants have been investigated with proven success. The new concept provides formation of more nucleation sites in nodular iron in addition to those initially generated by the magnesium-based ferrosilicon inoculant. Nodular irons from this treatment have been found to improve casting performance, microstructure and properties to a greater extent than other ferrosilicon inoculant alloys.

In this work, the effects of multiple inoculations and type of inoculants on the microstructure and the mechanical properties of nodular iron are investigated.

## 2. Experimental procedure

### 2.1. Materials

The ductile iron samples used in this study were produced by melting grey cast iron in a rotary furnace. The furnace was pre-heated for one hour before the pellets of grey iron along with 2 wt. % graphite were charged and then further heated for another one hour to ensure complete melting of the charge into liquid state. Some of the molten metal is poured into a ladle with a 2 wt. % FeSi inoculant placed at its base and subsequently cast in a mold to produce the ordinary grey cast iron. The ductile iron was produced by pouring the remaining molten metal into another ladle containing 5 wt. % Mg<sub>6</sub>FeSi nodularizer and 2 wt. % FeSi for the primary inoculation. Some of this was cast without any further treatment while some were subjected to further inoculation treatments with either 0.2 wt. % FeSi or 0.25 wt. % NiFeSi placed at the base of the sprues in the molds before pouring. Accordingly, four different samples, A, B, C, and D were prepared as shown in Table 1.

The cast samples were allowed to cool in the molds before they were removed, cleaned and fettled. The chemical compositions of the samples were determined using an ACL FISON Spectrometer, Model 3460, with a metal analyzer coupled to Microsoft Windows 2000 Software.

**Table 1:** Experimental Samples

Material	Description
A	Melted and recast grey iron
B	Nodularised cast iron using 5 wt.% MgFeSi and 2 wt.% FeSi for primary inoculation
C	Nodularised cast iron using 5 wt.% MgFeSi and 2 wt.% FeSi for primary inoculation and using 0.2 wt.% FeSi for secondary inoculation
D	Nodularised cast iron using 5 wt.% MgFeSi and 2 wt.% FeSi for primary inoculation and using 0.25 wt.% NiFeSi for secondary inoculation

## 2.2. Chemical and microstructural analysis

The chemical compositions of the samples were determined using an ACL FISON Spectrometer, Model 3460, with a metal analyzer coupled to Microsoft Windows Software. A 10 mm thick specimen was cut out of each of the samples A, B, C and D and carefully identified. The samples were in turn prepared for microscopic examination. Initial polishing was done on emery cloth with silicon carbide grade 0.1 and final polishing to mirror surface finish was with silicon carbide grade 0.3. Etching was done with Nital (2% Nitric Acid, 98% Alcohol). The samples were examined using an Optical Metallurgical Microscope with a camera attachment and photographs of the microstructures were taken at various magnifications.

## 2.3. Mechanical properties

For the determination of hardness, specimens were prepared to mirror surface on one end as described for the microstructural investigation (section 2.2) and the testing was conducted using the compression accessories on Mosanto Tensiometer Type W. The compression attachment was assembled together with compression die and a Brinell Ball Bolster. The mercury level was zeroed and with each specimen's polished surface held against the Brinell ball, the machine was loaded up to 750 kg (7358N). It was held in position for 15 seconds. On releasing the load and specimen taken out, the indentation value was read off with the Brinell reading microscope. Three indentations were made on the same surface of each specimen in turn and were averaged. The Brinell Hardness Number (BHN) was then read off from the appropriate set of tables. The ultimate tensile strengths (UTS) from the Brinell hardness number were approximated using the relationship in Equation (2) [5].

$$UTS = 3.4 \times BHN \quad (2)$$

## 3. Results and discussion

### 3.1. Chemical compositions

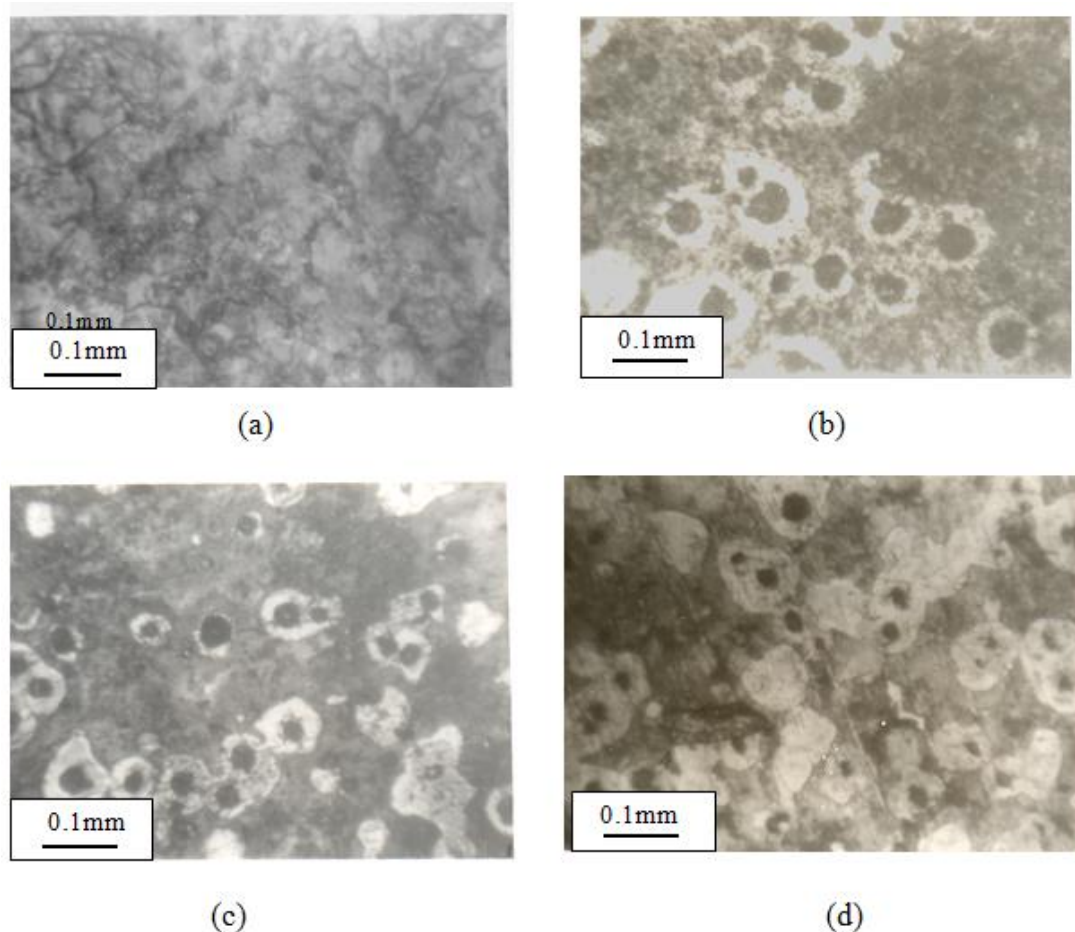
Results of the chemical analysis are shown in Table 2. Specimen A is the base material in this investigation. The carbon and silicon contents (>3%) of the cast irons are relatively significant. Other elements are in trace amounts except in the nickel-bearing ferrosilicon inoculated ductile iron (specimen D) with 0.67% nickel. The carbon content in specimens B and D (3.56% and 3.49% respectively) shows increment but decreases in specimen C (3.09%) where the silicon content is on the higher side. This is a specification for high strength cast iron [5]. There is an increase in the silicon content of the specimens B (3.79%), C and D (3.74%) when compared with its content in the base material but specimen C (3.68%) has a little less than others. The sulphur and Phosphorus contents decrease in specimens B, C and D (0.04% and 0.03%, 0.04% and 0.02% and 0.06% and 0.02% respectively) when compared with A (0.08% and 0.04%). The production of ductile iron necessitates a decrease in sulphur content [4, 5]. The Nickel content in specimens B and C decreases but shows increase in specimen D. Nickel enhances finer nodules, uniform graphitisation and yield softer iron matrix [5]. Chromium is lower and same for specimens B, C and D, tin is higher and same for the three specimens compared to A while Mo and Cu are constant for all the samples. Other elements are in very trace amounts. The effects of silicon and manganese on graphitisation can be observed in the increase in carbon content of specimens B, C and D. Increase in the silicon content of these three specimens are due to the inoculants (FeSi and NiFeSi) used.

**Table 2:** Chemical Compositions of Nodular Iron Samples

Specimen	C	Si	S	P	Mn	Ni	Cr	Mo	V	Cu	Sn	Al	Fe
A	3.28	3.15	0.09	0.04	0.40	0.03	0.08	0.01	0.01	0.04	0.02	0.00	Bal
B	3.57	3.79	0.04	0.03	0.44	0.02	0.07	0.01	0.00	0.04	0.03	0.00	Bal
C	3.09	3.68	0.04	0.02	0.44	0.02	0.07	0.01	0.00	0.04	0.03	0.01	Bal
D	3.49	3.74	0.06	0.02	0.39	0.66	0.07	0.01	0.00	0.04	0.03	0.01	Bal

### 3.2. Microstructures

Figure 1 shows the optical micrographs for the materials A, B, C and D. The microstructures of the ductile irons B, C and D (Figs. 1b, c and d) show graphite nodules which are absent in the grey cast iron A (Fig. 1a). They are similar to typical structures of grey and nodular cast irons. Samples with multiple inoculants show microstructures with finer and higher density of nodules (Figs. 1c and d.). The nodules are finer and more for the sample D with NiFeSi as the secondary inoculant (Fig. 1d) than for that (specimen C) with FeSi as the secondary inoculant (Fig. 1c).



**Fig. 1:** Microstructures of (a) Grey cast iron, (b) Ductile iron with single inoculation (c) Ductile iron with FeSi as secondary inoculant, and (d) Ductile iron with NiFeSi as secondary inoculant.

### 3.3. Mechanical properties

Table 3 shows the Brinell hardness values and the calculated Ultimate Tensile Strengths for the various material samples. The hardness and the ultimate tensile strength of grey cast iron, A (170 and 578 respectively) are lower compared to the ductile irons B (207 and 704), C (255 and 867) and D (187 and 636). It is seen that the values for specimen C (with 0.2% FeSi as secondary inoculant) are higher than other samples. This is because the graphite nodules of specimen C are more and finer than those of Specimen B (with 2% FeSi primary inoculation only), as shown in Figs 1b and 1c. This observation is in agreement with the established fact that more grain boundaries resulting from greater nodule density favors higher strength [4]. Furthermore, the composition showing carbon content (3.09%) on the relatively lower side of the range and silicon (3.68%) on the higher side is designed for higher strength [5]. Values for specimen D, with NiFeSi as secondary inoculant are lowest for the ductile irons despite that its nodules are the finest and even lower than those of specimen B with a single inoculation treatment. Greater hardness was expected of specimen D due to the reasons that are responsible for the improved hardness and strength of specimen C. However, the nickel in this specimen D (in the NiFeSi secondary inoculant), like silicon, serves as a graphitizer and as a result could have depleted the cementite in the matrix of some carbon [5]:



The carbon is precipitated as graphite, which is a soft material, and so, the effect is a lower hardness as observed in the case of specimen D.

**Table 3:** Hardness and Tensile Strengths of Samples

Specimen	Hardness (BHN)	Tensile Strength (N/mm <sup>2</sup> )
A	170	578
B	207	704
C	255	867
D	187	636

## 4. Conclusions

The investigation of the effects of multiple inoculation on the microstructure and mechanical properties of nodular iron has shown that:

- 1) Multiple inoculations refine graphite nodules thereby increasing the nodule count.
- 2) Graphite nodules refinement by multiple inoculations with ferrosilicon as the primary and secondary inoculant is accompanied with higher hardness and tensile strength.
- 3) Multiple inoculation with ferrosilicon as the primary inoculant but Ni-ferrosilicon as the secondary inoculant produces finer nodules and higher number density.
- 4) Despite the finer and higher nodule count with Nickel-ferrosilicon as the secondary inoculant than for ferrosilicon, the hardness and tensile strength values are lower.

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