The role of inoculant on grey cast iron (GCI) and impact on microstructure and mechanical properties

Pertunia Seema[1*], *Desmond* Klenam[1], *Michael* Bodunrin[1], *Christo* Hattingh[1], *Joseph* Moema[2] *and Maje* Phasha[2]

[1]University of Witwatersrand, Faculty of Engineering, School of Chemical and Metallurgy
[2]Advanced Materials Division, Mintek, 200 Malibongwe Drive, Randburg, 2194, South Africa

Abstract. This research was conducted to explore the influence of inoculants on the microstructure and mechanical properties of grey cast iron. The study also reviews the literature related to graphite morphology, solidification behaviour, and its impact on mechanical properties. However, inconsistencies in reported work highlight the need for a standardised approach to the inoculation of cast iron. Previous studies reported the successes and limitations of these inoculants on grey cast iron identifying the need to explore alternative inoculants such as FeSi-Zr. This study explores the effect of FeSi75-Zr inoculant on the microstructure, thermal and mechanical properties of grey cast iron. FeSi75-Zr inoculants were added at varying quantities of 0.1%, 0.2%, and 0.4% into grey cast iron materials. Additions of inoculant increased eutectic temperature and promoted the formation of short, sharp graphite flakes within the matrix. Notably, the addition of 0.2% FeSi-Zr inoculant resulted in the most refined microstructure and a hardness value that decreased from 172HV of un-inoculated to 156HV of 0.2% inoculanted. Increasing inoculation content led to an increased ultimate tensile strength (UTS) of 229 MPa.

1 Introduction

Cast iron is a versatile engineering material whose properties have evolved over the past years in line with improvements in the field of material science and technology. As from 2018, the various forms of cast iron accounted for 70% of the 110 million tons of total metal cast per year worldwide (10% for cast steel, and 20% for aluminium and other alloys) [1]. Cast iron is a class of iron-carbon alloys that consist of carbon content ranging between 2%C - 5%C [2]. One of the most widely used cast iron to date is grey cast iron (GCI) due to its favourable mechanical properties. GCI is a broad class of ferrous casting alloys distinguished by a microstructure of flake graphite in a ferrous matrix. It is primarily a Fe–C–Si alloy containing small quantities of other alloying elements such as sulphur, manganese, and phosphorus [3]. GCI is also the most widely used casting alloy due to its properties. It is classified into 5 categories: Type A, B, C, D and E based on graphite distribution, size and

* Corresponding author: 1431415@student.wits.ac.za

shape [4]. Type A, which is characterised by evenly distributed short, sharp flake-like graphite is mostly a preferred option due to its superior mechanical and microstructural characteristics.

The quality of GCI is dependent on its temperature control during solidification. It is therefore deemed important to understand the solidification structural morphology of the material to achieve the desired properties. The solidification begins with nucleation of the nucleaus or grain embryo, followed by nucleus growth unti it forms a grain within the matrix [3]. The number of nuclei in the molten metal affects the degree of graphitisation and eutectic cell count. Graphite formation is favoured at a eutectic temperature of 1147 °C or above [3]. If the temperature drops below 1147 °C, the rate of carbide formation becomes higher than that of graphite formation [3]. The introduction of silicon in the molten metal increases the eutectic temperature by 6 °C which reduces the carbide formation and promotes graphite nucleation [4].

Inoculation is widely recognized in the foundry industry as a standard metallurgical practice for improving the microstructure and mechanical properties of cast irons. Numerous studies confirm that FeSi-based inoculants containing elements like calcium (Ca), barium (Ba), strontium (Sr), zirconium (Zr), and rare earth metals are routinely added during cast iron production to promote graphite nucleation and decrease carbide formation [1][45][11]. There are a variety of commercial inoculants that are used in cast iron according to the desired properties of the materials. Literature indicates that FeSi-based inoculants have proven to be more effective and have largely been adopted within the foundry industry [19][20]. The study aims to evaluate the effectiveness of zirconium-containing Ferrosilicon inoculant on the microstructure and mechanical properties of GCI. Although Zr is known for its nucleation on GCI, its role in GCI inoculation remain insufficiently studied compared to other inoculants such as Ba and Ca. Additionally, it seeks to determine the optimal inoculant dosage for GCI production. The microstructural and mechanical analysis of the grey cast iron samples inoculated with varying additions of Sr and Zr was correlated with the solidification curve generated using the ATAS MetStar version 10.4 system.

2 Methodology

Before commencing with experimental work, a brief literature review on inoculation, thermal analysis, microstructural characterization, and nucleation methodology was conducted. The influence of inoculation on GCI was analysed. The search engine used are google schoolar, scopus. The keywords focused on was inoculation,grey cast iron production and Ferrosilicon with Zirconium. From the information provided by relevant studies choosen, the gap identified.

2.1 Materials and methods

Pig iron, GCI remelts, ferromanganese, pure silicon, and FeS were added into a 150kg capacity induction furnace. The batch was heated up to 1400 °C and held at that temperature for at least 10 minutes. Ferrosilicon-based inoculant with 75% silicon, containing zirconium was prepared with the particle size of 1.2 mm. The first melt, being the control sample, was tapped into the ladle without inoculation [21]. The addition of 0.10 wt.%, 0.20 wt.% and 0.40 wt.% of the inoculant was introduced to the metal stream of the second, third and fourth melts respectively to produce three different inoculated GCI samples. The selected grade of inoculant for ladle inoculation was added to the metal stream during tapping from the furnace to the transfer ladle. The molten metal was then poured into the sand moulds. Both the control and inoculated samples were given adequate time to gradually transform into solid castings at room temperature (23 °C) inside the mould. Optical emission spectrometry was used to analyse the chemical compositions of the GCI samples.

2.2 Thermal analysis

To collect thermal data and to identify the effect of inoculants, the molten metal was first poured into an alumina thermal cup fitted with thermocouples and analysed by ATAS (Adaptive Thermal Analysis System) MetStar version 10.4 system as indicated in Figure 1. MetStar system is designed for the cast iron production process, and it is utilized for recording solidification curves, controlling batching quantities, calculating carbon equivalent percentages, and predicting mechanical and microstructural properties of the casts.

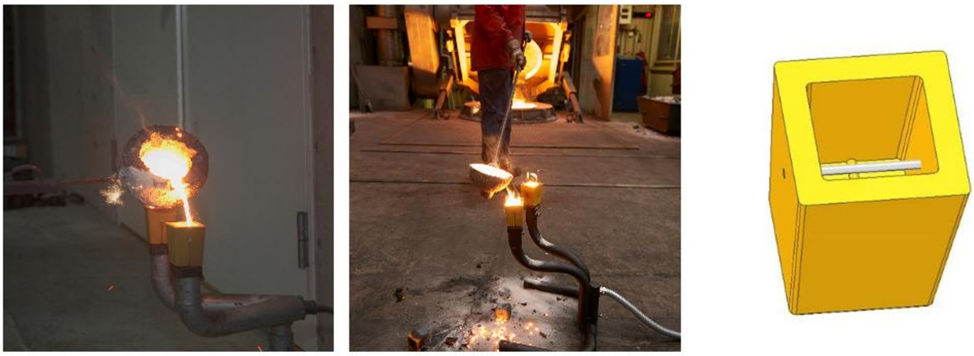


Fig. 1. Thermal analysis cup without tellurium, cup heads to place the thermal analyses cup at on the sampling stand.

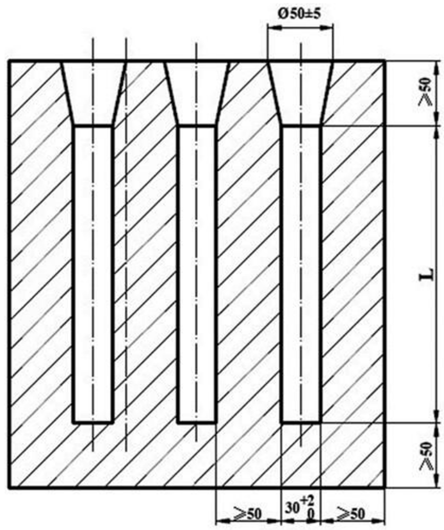
2.3 Microstructural examination

Metallography specimens were removed from the keel blocks and prepared based on ASTM E3-11 standards. Samples were consecutively ground on water-lubricated silicon carbide abrasive papers of 200 - 1200 grit sizes and polished to 1 μm mirror surface finish using diamond suspension polish. These were analysed in the polished condition and subsequently etched using 2% nital for detailed microstructural observations. Image J was used to determine the graphite count within the microstructure as well as the graphite size.

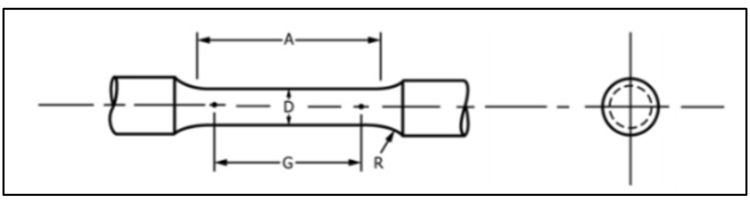
2.4 Tensile and hardness testing

The test bars from which the tensile test piece was machined were cast as a uniform cylindrical bar of 30 mm diameter and 300mm height, shown in Figure 2(a). The tensile test was carried out per BS 18: Part 2 standard. A tensile test specimen conforming to the dimensions given in Figure 2(b) was used.

Vickers hardness tester with a 10kgf load was utilised to perform hardness measurements. The samples were ground and polished to ensure flat and smooth surface for consistent and accurate indentation. Five indentations were made on each sample, and the average value was reported.



(a)



(b)

Fig. 2. (a) GCI test bar mould sketch and (b) machined tensile test specimen (plain ends) [18].

Table 1: Dimensions used for tensile testing samples.

A – Length of reduced section	57.2 mm
D - Diameter	12.7 mm
G- Gage length	50 mm
R – Radius of fillet	9.5 mm

3 Results and discussion

3.1 Literature review

The demand for GCI has been steadily increasing because of its affordable price and wider use in various structural components. The automotive sector, a major consumer of metal castings, faces the challenge of ensuring quality due to metallurgical defects. Studies have shown that castings using compound inoculants exhibited increased graphite content, enhanced matrix uniformity, and improved machinability compared to those treated with single inoculants [6][9]. Elevated graphite levels and consistent microstructural homogeneity may enhance the machinability of grey iron castings by reducing tool wear and cutting forces during machining [6].

The literature showcase the influence that FeSi inoculants have on GCI production. The unique combination of strength, corrosion and wear resistance, thermal conductivity, machinability, vibration damping capacity and castability afford the material a wide range of industrial applications [6].

In foundry operations, inoculants introduced to molten iron before casting impact primary structure features like austenite, carbides, eutectic cells, and graphite, and indirectly affect the eutectoid structure, particularly the pearlite/ferrite ratio, which relies on the quantity and shape of graphite. The advantages of inoculation of castings are numerous, some of these, for GCI, are (i) chill reduction (ii) graphite formation promotion, (iii) formation of fine graphite reduction (iv) uniform structures in various section promotion and (v) mechanical properties and machinability improvement [7].

Kutelu examined the effect of different percentages of ferrosilicon (FeSi) addition on the microstructure and mechanical characteristics of GCI [7]. The microstructure of the control sample (0.00 wt. % FeSi) displayed primary dendrites and graphite flakes, whereas the microstructure of FeSi inoculated samples showed a greater amount of more advanced primary dendrites, elongated graphite flakes, and austenite dendrites as illustrated in Figure 3(a-e). Based on the tensile results shown in Figure 4, the elevated tensile strength values of samples b, c, d, and e compared to sample a (see Figure 3) can be linked to the influence of ferrosilicon addition. However, the study did not examine FeSi-Zr-based inoculant which will be the focus of this research. While prior studies have mostly studied graphite morphology qualitatively, this study will also shed light on the stereological analysis (flake size, count, and type) using ImageJ software to assess microstructural quantitatively.

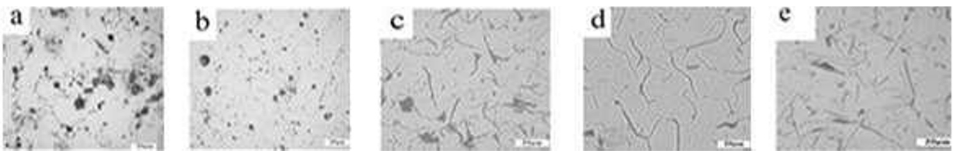


Fig. 3. Optical microstructures of (a) Control, (b) 0.50 wt. %, (c) 0.10 wt. %, (d) 1.5 wt. % and (e) 2.00wt. % FeSi inoculated GCI samples at Mag. 200X [7].

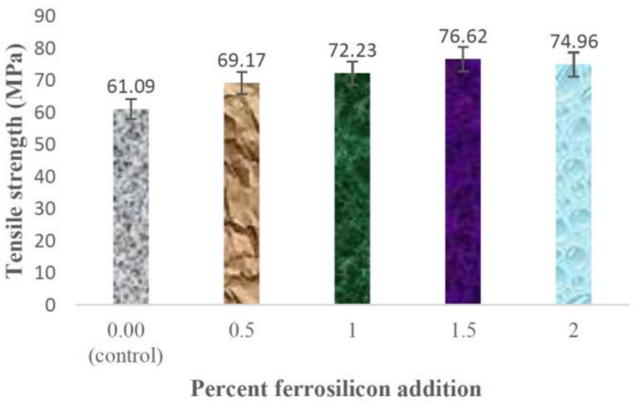


Fig. 4. Tensile strength of (a) Control, (b) 0.50 wt. %, (c) 0.10 wt. %, (d) 1.5 wt. % and (e) 2.00wt. % FeSi inoculated GCI samples [7].

3.2 Chemical composition

From the compositional data shown in Table 1, silicon content increases from 1.42% in the un-inoculated material to 1.91% in the 0.4% inoculated sample, which correspondence with the amount of ferrosilicon added.

An introduction of Si into GCI increases the eutectic temperature and improves both graphite morphology and mechanical properties [3]. Although Seidu reported that increasing Si content decreases carbon content, the correlation between the two was not observed in our data [17]. Each element observed in Table 2 has its primary metallurgical functions which are discussed below:

- Si improves graphite nucleation because Si is a graphite promoter [3].
- Al, Zr, Nb, and Ni refine the graphite into finer short graphite flakes [8,10,11].
- The S reacts with Mn to form MnS for improvement of pearlite formation [15].
- Copper and molybdenum support pearlite and carbide stability which increases wear resistance [12,15].
- Chromium improves materials' hardness and wear resistance but reduces graphite formation [13].
- Phosphorus improved fluidity during the casting process [14]

The chemical composition that was analyses and presented on Table 2 below indicate different elements in the GCI that have different benefits as indicated above. The presence of elements such as Si, Al, Zr, Nb, and Ni contributed to the refinement of the microstructure, leading to an increased graphite count and the formation of shorter, finer flakes with increasing inoculant addition. Additionally, the presence of S, Mn, Cu, and Cr played a role in stabilising the carbides within the microstructure.

Table 2: Chemical compositions of the control and inoculated GCI samples (wt.%).

	Controlled Sample	**Inoculated Samples**		
Elements wt.%	**Un-inoculated**	**0.1%Zr**	**0.2%Zr**	**0.4%Zr**
C	3.33	3.57	3.5	3.92
Si	1.42	1.51	1.55	1.91
Mn	0.59	0.59	0.58	0.56
P	0.032	0.037	0.035	0.035
S	0.096	0.092	0.085	0.055
Cr	0.07	0.073	0.067	0.035
Ni	0.03	0.028	0.029	0.026
Mo	0.034	0.004	0.0041	0.0045
AI	0.0071	0.0064	0.0053	0.0082
Cu	0.016	0.0028	0.0067	0.014
Nb	0.0012	0.0017	0.001	0.001
Zr	0.0015	0.0021	0.0036	0.0086

3.3 Thermal analysis

Inoculant addition has a significant impact on solidification curves. The solidification morphology for the metal begins with nucleation followed by nucleation growth. Nucleation is influenced by the number of nuclei present within the melt. Through the investigation, it was discovered that an increase in nuclei promotes graphitization and eutectic cell count thus affecting solidification curves [6].

Figure 5 demonstrates how each temperature was impacted as inoculants quantity increased. Liquidus temperatures (TL) represent the point at which austenite formation begins. From the experiment, it was observed that the un-inoculated and the first two samples of inoculated material (0.1% and 0.2%) have similarities (TL). However, the sample with 0.4% inoculation had a much lower (TL) in comparison to others. This finding can be attributed to nuclei formation rate which is dependent on the quantity of the material. A high nucleation formation rate can drop the temperature significantly before solidification starts.

The (TL) has a significant impact on undercooling (ΔT) as shown in Figures 6 and 7. Undercooling (ΔT) represents the difference between liquidus temperature (TL) and eutectic temperature (TELow). From the investigation conducted by Chisamera which was mainly focused on inoculation methods (in-ladle and in-mould inoculation) and how they impact the solidification of the material, it was discovered that lower undercooling (ΔT) accelerates the transformation of austenite to eutectic cells. This indicates that the decrease in (ΔT) improves graphite formation within the microstructure [7, 16].

Un-inoculated samples in Figure 6 show lower eutectic temperature (TELow) while inoculated melts shift the temperature upwards. Eutectic temperature (TELow) represents the point at which austenite starts to transform into eutectic cells [8]. Bangane investigated the effect of different inoculants which are FeSi based on the solidification of ductile iron [23]. The general findings showed that inoculation addition increases the (TELow). Minkoff recommends the eutectic temperature (TELow) of 1147 °C or above to suppress carbide formation [3]. The actual eutectic temperatures (TELow) for all samples in this research fall below 1147 °C but graphite formation was not affected as indicated in Figure 8. According to Metstar software settings, the range for eutectic temperature that favours graphite formation falls between 1118.9 °C and 1136.5 °C which is consistent with our experimental results. From Figure 6, it can be observed that the (TELow) is consistently increasing as inoculants addition increases.

Higher eutectic temperature (TEHigh) represents the point at which eutectic cell formation and growth is completed [8]. The difference between the (TELow) and (TEHigh) is labeled as a recalescence (R) [16]. (R) is a release of latent heat during the eutectic growth. Smaller recalescence favours graphite formation [16]. Figure 7 shows a decrease in recalescence as inoculants are added and the recalescence of 0.4% inoculated GCI is barely existing with a value of 1.8 °C. (TELow) contribute significantly to a reduction in recalescence. Solidus temperature (Ts) represents the point at which the material has solidified into a complete microstructure which marks the end of the solidification process.

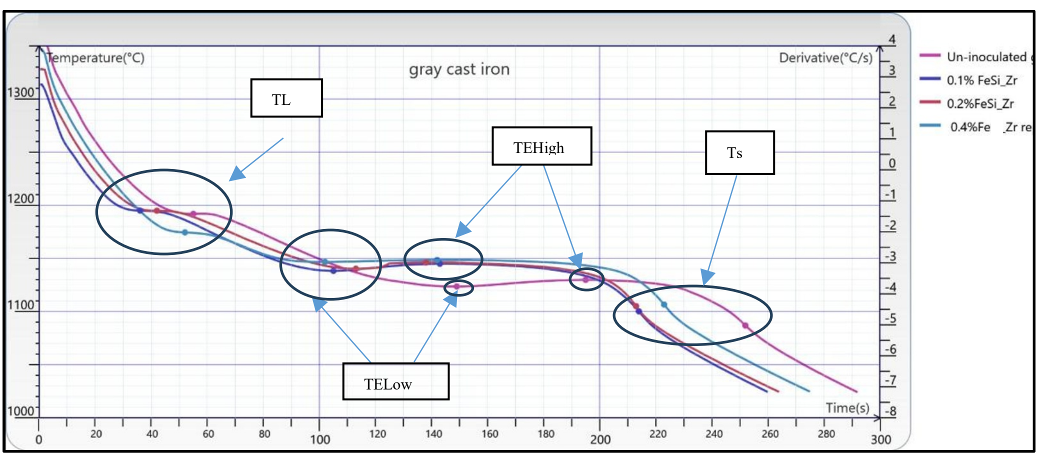


Fig. 5a. Solidification curves obtained from MetStar software for control and GCI samples inoculated with 0.1% FeSi-Zr, 0.2% FeSi-Zr, and 0.4% FeSi-Zr.

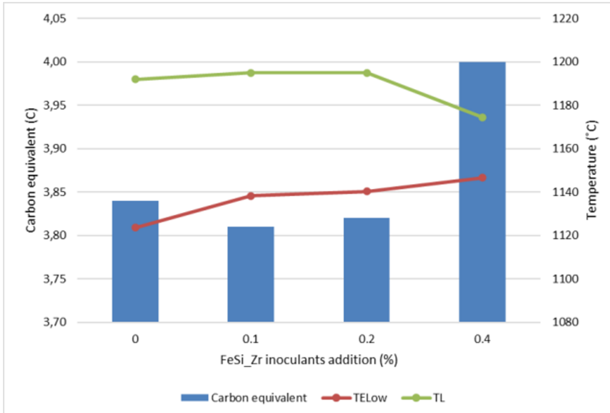


Fig. 6. Carbon equivalent, eutectic temperature and liquidus temperature for GCI samples with inoculants addition of 0.1% FeSi-Zr, 0.2% FeSi-Zr and 0.4% FeSi-Zr.

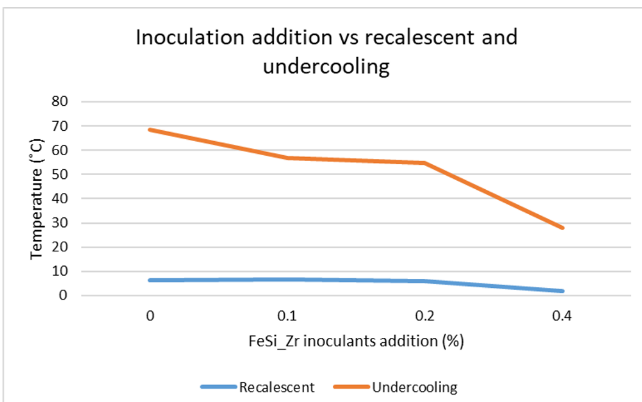


Fig. 7. Recalescence and undercooling for GCI samples with inoculants addition of 0.1% FeSi-Zr, 0.2% FeSi-Zr and 0.4% FeSi-Zr.

The solidification behaviours and formation of graphite in cast irons are not only influenced by inoculation practices and temperature but also the carbon equivalent (CE). Equation 1 demonstrates the carbon equivalent calculation method.

$$\mathbf{CE = \%C + 0.33(\%Si) + 0.33(\%P)} \qquad \textbf{(Equation 1)}$$

From Figure 6, carbon equivalent ranges between 3.81% and 4.00%. The addition of inoculants influenced the carbon equivalent hence it shows that an increase in inoculants increases the carbon equivalent. Based on research conducted, it can be observed that GCI is produced with a carbon equivalent that is below 4.00%. Even though the hypoeutectic GCI states the maximum at 4.3%, an approximation of 3.80% to 3.85% has been proven to be the most efficient.

Seidu conducted a study to observe the effect of shakeout time on the microstructure and hardness properties of GCI when subjected to different quantities of FeSi inoculants [17]. From the results, carbon equivalent increased proportionally with inoculant addition levels [17]. This correlates with the applicability of our research.

3.4 Microstructural analysis

Inoculation addition improves the metallography of the material by increasing the graphite quantity and refining the size of the graphite flake. Figure 8 illustrates a gradual increase in graphite as more inoculants are added. Kutelu also examined the effect of inoculants on GCI as the addition percentage increases [7]. From the finding, the graphite amount increased as inoculants increased but the optimum addition was 1.5% which is extremely high. The difference between these addition control and the author's is the quality of the ferrosilicon. The ferrosilicon added did not have any special elements such as Zr, Sr, or RE metal. This shows the effectiveness of special elements on the material. By looking at Figure 8, the addition of 0.2% FeSi has a noticeable increase in graphite number. In comparison to B and A, the graphite sizes are smaller.

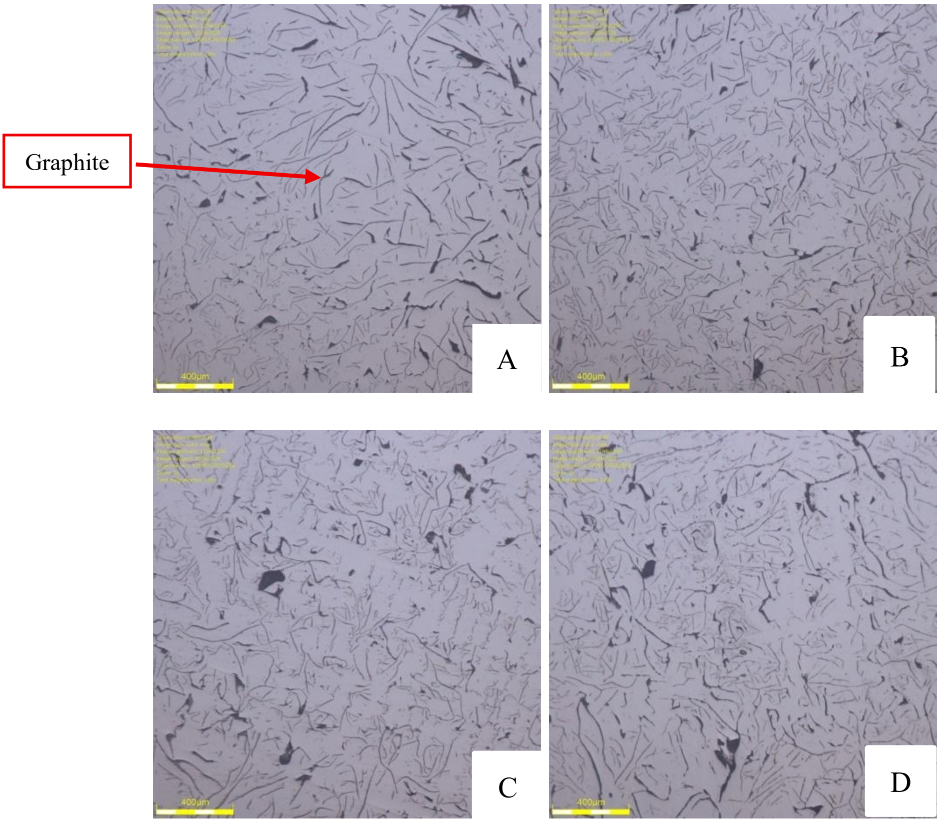


Fig. 8. As polished microstructures of control and inoculated samples of GCI at a magnification of X139 - (A) control (un-inoculated), (B) 0.1% FeSi-Zr, (C) 0.2% FeSi-Zr and (D) 0.4% FeSi-Zr.

To determine the actual amount of graphite within the matrix, image J was utilized. From the analysis shown in Figure 10, un-inoculated GCI contains 1846 graphite with an average area of 66μm^2 per graphite whereas else that of 0.2% FeSi addition contains 2716 graphite with an average area of 50μm^2/graphite. After the addition of 0.2% FeSi, the size of the graphite becomes constant and the inoculation quantity decreases. This suggests that 0.2% FeSi addition is the optimum amount.

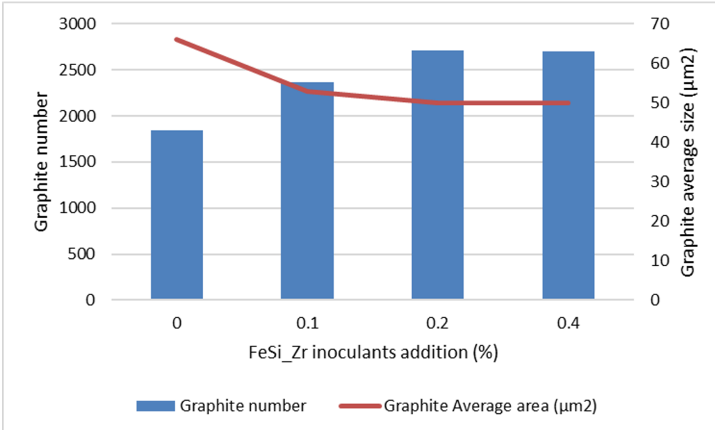
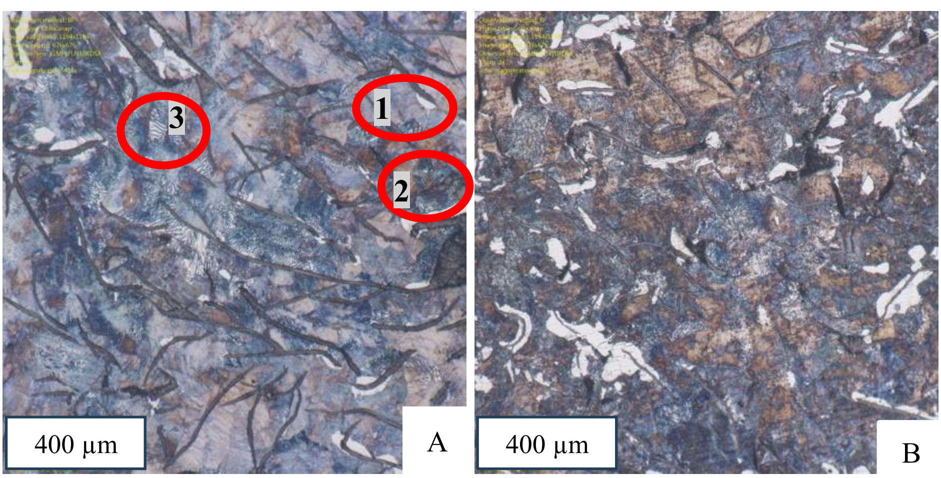


Fig.9: Graphite number and average size for control and inoculated GCI - 0.1%Zr, 0.2%Zr and 0.4%Zr.

Figure 10 illustrates etched cast samples. An etchant is used to highlight phases within the microstructure. GCI has three different phases within the matrix which are graphite, ferrite, and pearlite. As indicated in Figure 10, graphite is represented by the number 1 which shows a flake-like black structure. Ferrite is represented by the number 2 which shows the light phase surrounding graphite. Pearlite is represented by the number 3 which shows the lamellae phase.

As inoculants are added, pearlite decreases. This results from an increase in graphite within the matrix. Both pearlite and graphite require carbon for their formation [3]. With high graphite content, pearlite will not have enough carbon to increase its phase, hence it decreases.



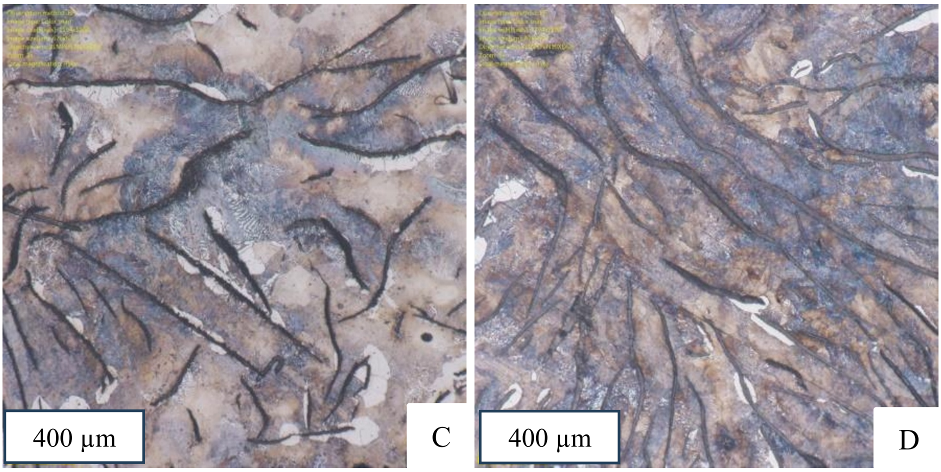


Fig. 10. Etched microstructures of control and inoculated samples of GCI at a magnification of X139. (A) control (un-inoculated), (B) 0.1%Zr, (C) 0.2% Zr and (D) 0.4% Zr

3.5 Tensile and hardness properties

Figure 11 shows that the hardness of GCI decreases from 172HV of un-inoculated melt to 132HV for 0.4% inoculation. This results from the graphite formation which decreases carbide and increases ferrite formation [6][7][11][22]. Lower hardness improves machinability, thermal shork resistance and damping capacity. However, wear resissance get reduced. The results obtained from this research correspond with findings from literature [17]. The hardness value of their findings was consistently reducing as more inoculants were added.

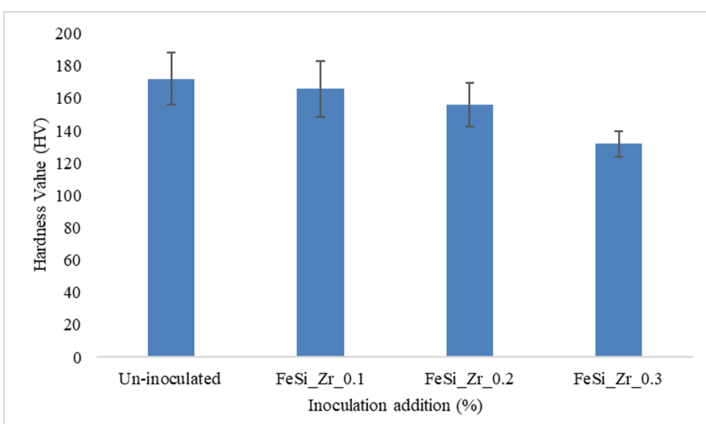


Fig. 11. Vickers hardness test of control and inoculated GCI samples (0.1%FeSiZr, 0.2%Zr and 0.4%Zr.)

The UTS for un-inoculated material was found to be lower than the inoculated samples. Increasing inoculation led to an increased UTS. This results from the effect of graphite size and distribution. Fig 8 and 9 show that increasing inoculants results in more graphite quantity and reduces and refines their size. This leads to more stress distributed evenly within the microstructure which allows matrix continuity and prevents immediate crack initiation.

The cooling rate also plays a significant role in UTS as it affects the microstructure. A faster cooling rate results in finer graphite hence it increases the UTS. NOVA has integrated UTS on their Metstar system based on cooling rate. Comparing the results obtained with the tensile test performed, their results are significantly higher than the actual value, but they are consistently increasing as expected but decreased on 0.4% addition. Based on estimated UTS by MetStar, 0.1% FeSi-Zr is the optimum addition with 268 MPa whereas else with actual testing, 0.2% FeSi-Zr is the optimum with the UTS of 229MPa. 0.4% FeSi-Zr addition led to a decrease in actual UTS which is the same trend with the MetStar UTS. The actual results obtained indicate that this material can be classified under the ASTM A48 Class 30 standard [18]. Observation made by Soiński (2011) shows that the addition of FeSi with Zirconium increases the UTS which relates to our findings. The research used 0.25% of FeSi-Zr for the experiment [9].

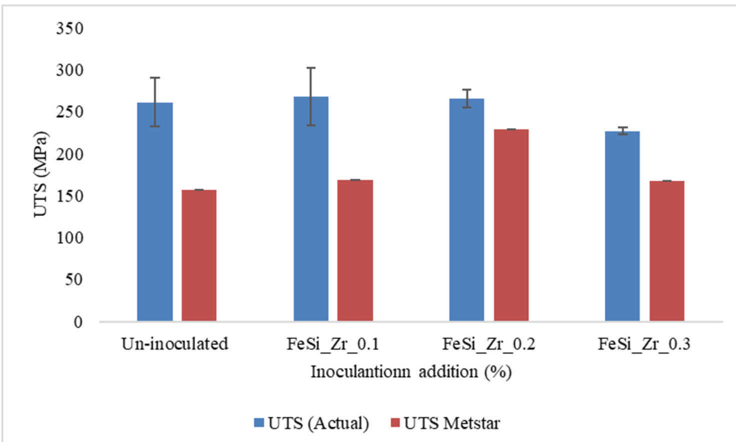


Fig. 12. Ultimate tensile strength of control and inoculated GCI samples (0.1%FeSi-Zr, 0.2% FeSi-Zr and 0.4%FeSi-Zr).

4 Conclusion

- This study has shown that the addition of inoculants improves the material's thermal, microstructural, and mechanical properties. However, when it start being in excess, the impact decreases noticebly. Looking at the results analysed, the optimum addition of inoculants to be considered is 0.2% FeSi-Zr due to the balance it presented between process thermal, microstructural, and mechanical integrity.
- The phase transformation temperatures along with graphite size, distribution, and amount for samples with 0.2% inoculants addition, have proven to be the optimum of all the samples.
- The pearlitic phase on the microstructure has been shown to decrease as more inoculants are added.
- The hardness value decreased with an increase in inoculants and UTS increased until it reached its optimum value of 228MPa.
- Inoculation improves GCI microstructure and properties, but discrepancies between MetStar predictions and actual results highlight the need for validation through physical testing. Further research on combined inoculants could enhance performance and reduce costs.

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Data Availability: Data supporting the findings of this study are available from the corresponding author upon reasonable request.

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