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Preconditiong and Inoculation of Low Sulphur Grey Iron

Md. Sojib S. Hossain *, A. K. M. Bazlur B. Rashid
Bangladesh University of Engineering and Technology
Shahbagh, Dhaka – 1000, Bangladesh
* Corresponding author. E-mail address: sojib.buet.bd@gmail.com

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Abstract

For quality grey cast iron production, the challenging issues are to avoid cementite structure and obtain the desired graphite morphology with proper matrix as well as hardness. The objective of the present research is to find out the right combination of preconditioner and inoculant that may help to overcome the challenges. In this work, sulphur content is kept low (0.01%). Two preconditioners namely metallurgical SiC and zirconium bearing FeSi with two types of inoculant are individually used to make four combinations of sample and for each case metal is poured into the green sand mould. Finally Brinell hardness and graphite morphology is observed in the thickest and thinnest portions of the castings. Metallurgical SiC with barium bearing inoculant gives better graphite morphology and hardness than strontium bearing inoculant, on the other hand zirconium bearing FeSi gives more satisfying result than SiC with every type of inoculant. Among all of the combinations Zr bearing preconditioner with Ba bearing inoculant gives good graphite morphology with best mechanical properties in both thickest and thinnest portions of the casting.

Keywords: Metallography, Mechanical properties, Preconditioner, Inoculant, Cementite

1. Introduction

In some special engineering applications where heavy machining, heat management, noise absorption and compressive strength are the vital issues, grey cast iron is the first choice. It is because, in quality grey cast iron free graphite are inherently distributed in the iron matrix. These free graphite act as a lubricant during machining hence increase machinability as well as heat conductivity, damping properties and compressive strength of the base iron [1]. The problem is, if the cooling rate and chemical composition are not properly synced or co-related to each other free graphite cannot be formed but hard & brittle cementite during solidification, ultimately grey cast iron loses its qualities [2, 3].

In this context metallurgists follow various melting and solidification process to suppress the cementite formation. The main concept of these process is to create proper nucleation sites for graphite precipitation so that undercooling temperature is decreased and solidification temperature is raised over the cementite eutectic and touched the graphite eutectic temperature [4]. Normally in base iron sulphur and Mn form MnS, itself MnS, various oxides, nitrides, and other non-metallic inclusions create core and (Mn,X)S compounds (X = Fe, Al, O, Si, Ca, Sr, Ti etc.) create shell of the nucleus sites. But these are not in hexagonal structure which actually graphite lattice structure conveys. This is the reason why various proprietary alloy (Ca, Sr, Ba, or Zr etc.) bearing FeSi is used as an inoculant or preconditioner. These proprietary alloy bearing FeSi forms hexagonal structure of very complex compound $XSiO_2$ or $XOAl_2O_3$ $2SiO_2$ where (X = Ca, Sr, or Ba) and these hexagonal structure cover the nucleus sites [5, 6]. Now hexagonal graphite can easily be precipitated on these complex hexagonal silicate compound.

The mechanism is not so easy that are explained here, it becomes more complex when grey cast iron is trying to produce from low sulphur refined steel scraps. As steel scraps are very clean and refined, melt produced from these scraps lacks in heterogeneous nucleus for graphite precipitation, so foundryman intentionally adds some preconditioner in furnace and inoculant in ladle or in stream condition. As MnS is a trusted nucleus site, most of the inoculant creates hexagonal phase very satisfactorily on it [7]. But when it is absent in steel scraps melt, use of the proper combination of other preconditioner and inoculant is mandatory for producing cementite free castings [8, 9].

This experiment is carried to find out the better combination of preconditioner and inoculant that will help to produce carbide or cementite free castings with desired hardness.

Although thousands of parameters are involved in foundry practice, cooling rate, casting geometry, chemical compositions, presence of nuclei, alloying elements etc. are the most important variables for cementite formation [10]. So improper preconditioner and inoculant or absence one of these obviously make a risk of cementite formation. Thin walled casting, faster cooling rate and various carbide former in the melt strengthen the risk. This experiment is conducted as an industrial assignment in the production line, so all the variables and foundry environment are more industry oriented so the result. Metallurgical SiC is a very popular and common preconditioner for grey cast iron, here we are trying to find out a right inoculant that works best in low sulphur environment with metallurgical SiC preconditioner. Again we are trying to quest another preconditioner where SiC cannot perform well.

In this experiment two preconditioners along with two inoculants are selected, so four combinations are obtained. Some pair gives better mechanical properties, some better graphitization, some are economically cheap, other give optimization of all properties. So foundryman can easily select their appropriate combination of preconditioner and inoculant after exploring this article.

2. Experimental procedure

Metal is melted in a medium frequency acid lined 2 ton capacity induction furnace, the charge recipe is 30% foundry return and 70% mild steel scrap. Chemical composition of these two charges are given bellow.

Table 1.
Chemical composition of the charge recipe

Elements (%)	Foundry return	Ms Scrap
C	3.2	0.2
Si	2.2	0.4
Mn	0.55	0.55
S	0.01	0.01

For first melt 1 % metallurgical SiC is used in the furnace as a preconditioner then 200 kg metal is tapped in a ladle and inoculated with inoculant B. Again 200 kg metal is tapped from the same melt, at this time metal is inoculated with inoculant I. In case of second melt 0.1 % zirconium bearing FeSi is used in the furnace as a preconditioner then inoculated separately with inoculant B and I following similar fashion as previously done. So four combinations of sample are found as follows.

Table 2.
Combinations of sample

Sl	Preconditioner	Inoculant	Sample name
1	Metallurgical SiC	B	1 B
		I	1 I
2	FeSi (Zr bearing)	B	2 B
		I	2 I

Metallurgical SiC is added with solid scraps in the furnace from the very beginning of the charging. On the other hand zirconium bearing FeSi is used during chemical composition adjustment and just before spectro analysis of the melt. The composition of two prconditioner is listed on the following table.

Table 3.
Composition of two preconditioner

Metallurgical SiC	FeSi (Zr Bearing)
SiC 90% (min)	Silicon 62-69 %
Free carbon 2.5% (max)	Zirconium 3-9%,
Fe_2O_3 2.5% (max)	Calcium 0.6-1.9%,
	Aluminium 3-5%
	Iron balance
Grain size 1-8 (mm)	Grain size 0-10 (mm)

In every test, furnace temperature is measured by Vsmart immersion temperature pyrometer after removal of the slag. When metal temperature is 1420°C-1460°C, chemical composition is measured and adjusted. After that ladle is prepared for tapping. During preparation, neutral refractory lined ladle is preheated by gas fired burner until the refractory of ladle looks red hot in colour. At 1450°C temperature metal is tapped and inoculated with 0.15% inoculant respectively. Inoculants specification are listed below.

Table 4.
Inoculant specification

Inoculant B	Inoculant I
Silicon 64-70%	Silicon 70-78%
Barium 2-3%	Strontium 0.6-1.2%
Calcium 1-2%	Calcium 0.1% (max)
Aluminium 0.8-1.5%	Aluminium 0.5% (max)
Iron balance	Iron balance
Grain size 2-7 (mm)	Grain size 2-7 (mm)

The holding time is 2-3min and the metal is poured in green sand moulds (around 2% new sand, 1% bentonite, 0.6% coal dust balanced is old sand). The physical properties of the green sand are, moisture content 3.5-4%, compactibility 40±2, active clay 10%, AFS clay 14%, wet tensile strength is 0.20 N/cm^2 bunch weight is 14 kg, pouring time is 6 sec, pouring temperature is 1350-1400°C and every crankcase (casting) weight is 1.6 kg which thickest and thinnest portions thickness are 45 mm and 15 mm.

3. Result and discussion

Before pouring the metal, coin sample is casted in a copper block then melt composition is analysed by "Spark Analyzer Pro LAB Version 1.04.0000 from SPECTRO Analytical Instruments GmbH'' The final composition of every melt is kept very close to the value listed on table 5.

Table 5.
Chemical composition of the samples

Elements name	Weight %
C	3.3
Si	2.3
Mn	0.6
S	0.01
Fe	93.7

a. Brinell hardness comparison

After the casting, crank cases are cooled naturally and the mould is broken down by cool drum. The castings are separated from the sand and shot blasted for final cleaning. Then hardness test specimens are prepared by Metkon METACUT 251 cutter in normal condition. After that Brinell hardness number on thickest and thinnest portion of more than 10 crankcase is measured by Affri Integral Hardness Tester following the ISO 6506 standard. For every sample 5 indentations are taken on each portion then average HBW is calculated for that sample. During test the room temperature is 24°C and average humidity is 40-50%. The ball diameter is 5 mm, test pressure is 750 kg and indentation time is 10 second. Finally average HBW of 10 samples are calculated and listed on table 6.

Table 6.
Hardness comparison

Sl No	Preconditioner	Inoculant	Sample name	HBW 5/750/10	
				Thickest portion	Thinnest portion
1	Metallurgical SiC	B	1B	191	235
		I	1I	196	329
2	FeSi (Zr bearing)	B	2B	210	230
		I	2I	178	221

As the cooling rate in the thinnest portion is faster than thickest portion, the eutectic austenitic dendrite is finer, as well as DAS and distance between two graphite is also small in the thinnest portion [11]. So the average hardness in the thinnest portion is higher than the thickest portion.

Inoculant B and I gives close result with zirconium bearing FeSi preconditioner (230 & 221 in thinnest portion and 210 & 178 in thickest portion). Inoculant I gives abnormal result with SiC for thinnest portion of sample 1I, hardness value is 329 HBW which indicates the different cast iron matrix rather than ferrite or pearlite, though with inoculant B metallurgical SiC gives very excellent hardness 235 in thinnest portion and 191 in thickest portion.

Although metallurgical SiC is cheap, it is needed in a bulk amount for proper preconditioning (1%) than Zr bearing FeSi (0.1%). Again using SiC increases the melting time, needs to use with solid charges otherwise carbon and silicon dissolution rate are decreased, moreover foundry returns become affluent of inclusion [12]. Now the point is, in industrial foundry for maintaining large volume of productions it is needed to re melt the foundry returns very frequently. In this case using metallurgical SiC as a preconditioner, the production cost need to be checked and process parameter should be optimised. So in both metallurgical and mechanical point of view, using Zr bearing FeSi rather than SiC may be a safer foundry practice.

Again, comparing with inoculant B and I hardness value of samples B (235 & 230 in thinnest portion and 191 & 210 in thickest portion) are fairly consistent and greater than I (329 & 221 in thinnest portion and 196 & 178 in thickest portion) within the technical limit. Inoculant B gives better result in the both portions, on the contrary inoculant I gives hard structure in the thinnest portion though acceptable hardness in thickest portions of the castings.

The better inoculant creates better environment for producing more eutectic cell and finer austenitic grains so better hardness [13, 14]. Again hardness value is proportional to tensile strength of the cast iron (same matrix) [15]. So inoculant B is observed as a better option than I for low sulphur grey iron environment on the basis of mechanical properties.

b. Observations and analysis of the microstructure

For microstructure observation the metallographic samples are prepared by Metkon FORCIMAT grinding and polishing machine.

Graphite morphology and matrix of the samples are observed by Nikon MA200 inverted metallurgical microscope and analysed by CLEMEX VISION PE software following DIN EN ISO 945 standard. The room temperature is 24°C and average humidity is 40-50%.

The microstructure of the produced low sulphur grey iron is presented in fig. 1-2, fig. 1 displays the microstructure in the thickest portion of the four samples making from four combinations of preconditioner and inoculant. Here the notation 1 signifies the metallurgical SiC and 2 signifies the zirconium bearing FeSi. In case of inoculant B indicates barium bearing inoculant and I indicates strontium bearing inoculant. Similarly in fig. 2 microstructure is presented of the same samples but thinnest portion of the castings. For matrix observation of the same samples in the same position, samples are etched with 2% nital and the metallographic images of thickest and thinnest portions are presented respectively in fig. 3-4.

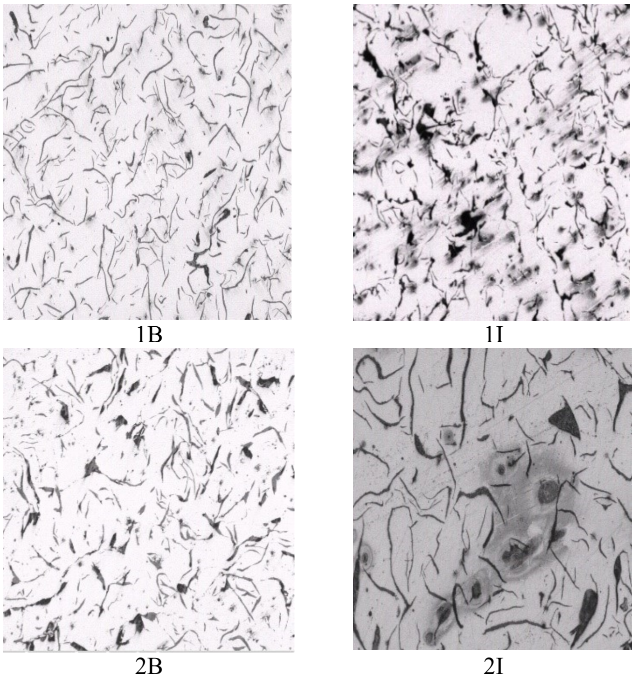

Fig. 1. Microstructure of the thickest portion of the samples (Mag.100x, unetched)

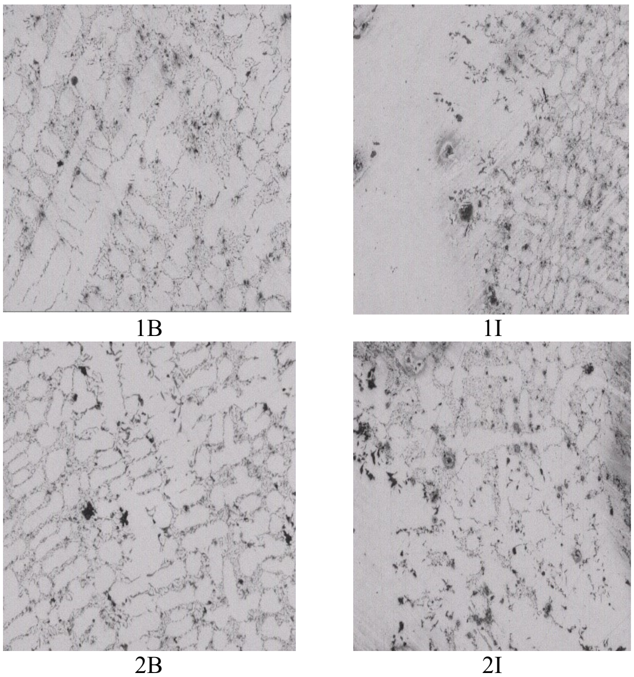

Fig. 2 . Microstructure of thinnest portion of the samples (Mag.100x, unetched)

Graphite distribution and size in both portion, at the same time total free graphite in the thinnest positions are calculated by CLEMEX VISION PE software. Here magnification is kept 100X and ISO 6506 standard is followed. Result is given bellow.

Table 7.
Graphite distribution and size %A(3-6)

Sl No	Preconditioner	Sample name	Graphite distribution and size (%A(3-6))	
			Thickest portion	Thinnest portion
1	Metallurgical SiC	1B	100	0
		1I	92	0
2	FeSi (Zr bearing)	2B	100	0
		2I	95	0

As the cooling rate is very slow in the thickest portion, graphite size and distribution is almost 100% A(3-6) in the thickest portion of all the castings. (Fig. 1 & table 7). This is the result of well preconditioning and inoculation of grey iron. There are many scientific evidence about the efficient preconditioning capability of metallurgical SiC as well as proprietary alloy bearing FeSi (16)

Due to high cooling rate, graphite distribution is basically D-type in the thinnest portion of the castings (Fig. 2 & table 7) and in some portions of the sample 1I free graphite are not clearly visible. Similarly for 2I free graphite visibility is not clear in some portions but graphite distribution on the other portions is clearly D-type. Unclear free graphite area may be an indication of poor inoculation effect and the matrix may be cementite or other carbides.

As all the visible free graphite are D-type in the thinnest portion, volume fraction of total free graphite is calculated to make a clear comparison among the four combinations. In this portions % free graphite is for sample 1B is 11%, 1I is 8%, 2B is16% and 2I is 10%.

Comparing the both types of pre- conditioner, performance of Zr bearing FeSi is noticed well (melt 2I & 2B) than metallurgical SiC in the thinnest portion with two types of inoculant. In this area % free graphite is higher and D type graphite are well distributed. Many researchers also concede the effectiveness of Zr bearing FeSi for avoiding cementite and D-type graphite formation [17]. Again for the samples of barium bearing inoculant (combination 1B & 2B) free graphite are more visible and well distributed than strontium bearing inoculant melt combinations1I & 2I.

Again for the comparison of matrixes, metallographic images of all the samples are magnified in 500X and phases are analysed by CLEMEX VISION PE software. Analysis result is displayed in the table given bellow.

Table 8.
Phase analysis (% pearlite)

Sl No	Preconditioner	Sample name	% Pearlite	
			Thickest portion	Thinnest portion
1	Metallurgical SiC	1B	89	98
		1I	94	73
2	FeSi (Zr bearing)	2B	98	97
		2I	93	79

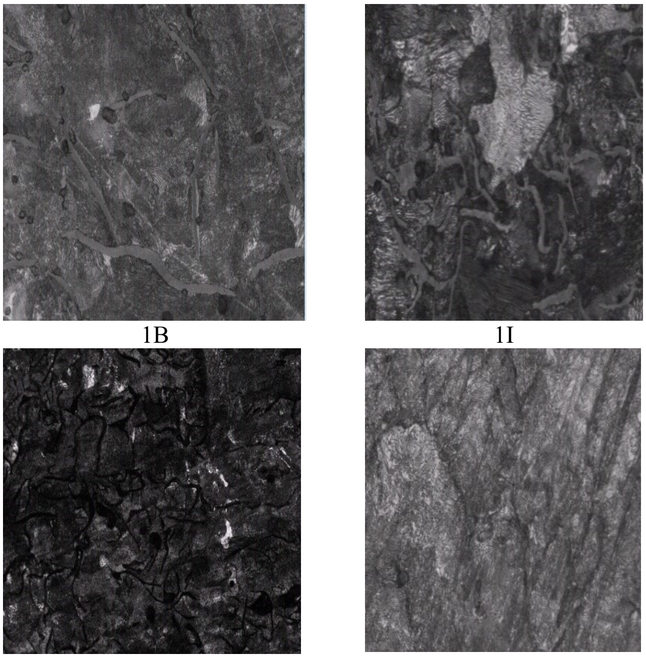

Fig. 3 . Matrix of the thickest portion of the samples. (Mag. 500x, 2% nital etched)

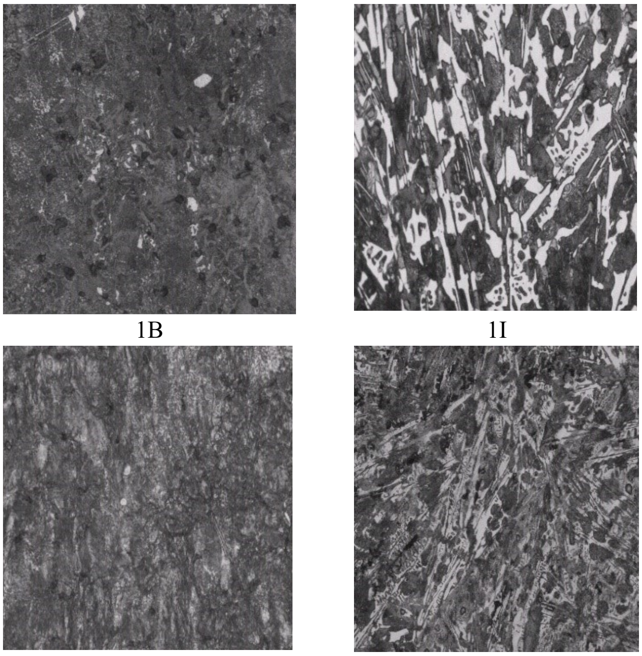

Fig. 4. Matrix of the thickest portion of the samples. (Mag. 500X, 2% nital etched)

Mechanical properties are greatly and in some cases machinability depends on the matrixes of the grey cast iron. So it is an important parameter of the quality grey cast iron.

In thickest portion all the matrixes are essentially pearlitic (Fig. 3 & table 8) % pearlite is more than 80%. Little fractions of ferrite are incorporated in the pearlitic matrixes. No carbides or cementite are visible especially in the thickest portions of the casting for all the samples.

In thinnest portion (Fig. 4) for both types of sample inoculated with inoculant I (melt 1I & 2I) bright white cementite or carbides are clearly visible and the % pearlite is not up to the mark 73% & 79%. On the other hand, matrixes in the same portion of the castings inoculated with inoculant B (melt 1B & 2B) is essentially pearlitic (more than 80%) 98% & 97%. If we think about the preconditioner, performance of the metallurgical SiC (melt 1I) is not as good as Zr bearing FeSi (melt 2I). The matrix of melt 1I in the thinnest portion is not essentially pearlitic (less than 80%) but hard cementite, though for sample 1B % pearlite is observed more than 80 in both thickest and thinnest portions. For Zr bearing FeSi (melt 2B & 2I) % pearlite is more satisfying than metallurgical SiC pre conditioner in case of (melt 1I).

4. Conclusions

In this experiment four combinations of grey iron sample are casted and investigated. These are casted with two preconditioner (metallurgical SiC and Zr bearing FeSi) and two inoculants (barium bearing FeSi and strontium bearing FeSi). Conclusions are noted bellow regarding the information attains from this current experiment.

It is necessary to add preconditioner and inoculant properly for producing quality grey cast iron from low sulphur mild steel scrap.

For thick casting (current experiment is 45 mm) all the four combinations are industrially applicable. Graphite size, distribution, amount of pearlite and hardness are very satisfying. On the basis of availability, cost, repeatability of foundry return melting and casting defects one can select any combinations of sample for producing low sulphur high quality grey cast iron.

In case of thin walled casting (current experiment is 15 mm) care should be taken for selecting preconditioner and inoculant. Metallurgical SiC with strontium bearing inoculant is the worst selection and Zr bearing FeSi with Sr bearing FeSi inoculant is the marginal option. Both two combinations of sample form more or less hard cementite structure and % free graphite is comparatively less.

So in low sulphur environment strontium bearing FeSi may be avoided but barium bearing FeSi inoculant is the best choice. Both metallurgical SiC and zirconium bearing preconditioners give excellent metallurgical and mechanical properties with barium bearing FeSi inoculant.

Literature suggests in some places that re melting of more and more metallurgical SiC contained foundry return may cause inclusion type defect and make the melting process slow. So zirconium bearing preconditioner along with barium bearing FeSi inoculant is noted as the best combination among all of the four samples for producing low sulphur high quality grey cast iron.

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