# STRUCTURAL CHARACTERISTICS OF RE-INOCULATED GREY CAST IRONS

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*The paper describes the characteristics of a hypoeutectic grey cast iron microstructure in the first ten minutes after inoculation and reports on the possibility of restoring the inoculation effects by re-inoculating after this period. A wedge type casting was evaluated to monitor the influence of cooling rate on the fade characteristics of the test iron. A second, lower inoculant addition in a late inoculation practice is recommended to restore most of the typical inoculation effects, such as fewer carbides and less undercooled graphite with associated ferrite*

**Keywords**: grey iron, inoculation, fading phenomena, graphite, carbides, matrix

## 1. Introduction

The most important objective of a reliable production of grey cast irons is to avoid free carbides formation, even at high cooling rates, solidifying with thin walls and sharp edges, to promote type A graphite formation to avoid undercooled graphite with a homogenous structure throughout the casting section. These requirements demand the effective nucleation of graphite, which means that the lowest eutectic temperature (TEU) must be higher than the metastable (carbidic) equilibrium temperature (Tmst). Also, TEU must be high enough to carry out the following condition [1, 2]:

$$\Delta T_1 = TEU - Tmst > 25^oC \quad (1)$$

Inoculation is essential in the production of all higher strength grey cast irons [3-5]. This metallurgical treatment of the liquid iron immediately prior to solidification is essential to promote enough nucleation sites for graphite, so that

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solidification occurs with less eutectic undercooling below the stable (graphitic) eutectic temperature to obtain a minimum 25°C for the $\Delta T_1$ parameter.

An inoculant is a material added to the liquid iron just prior to pouring into a mould to form a casting. Typically a FeSi based alloy is used, incorporating some active elements, such as Al, Ca, Ba, Sr, Ce, La, to provide a suitable phase to facilitate nucleation of graphite during solidification [5]. Even at a relatively low addition rate to the iron melt (0.05…1.0wt.%), this inoculating material can totally change the solidification pattern of cast iron.

Recently it was found that complex manganese sulphide (Mn, X)S type compounds, where X = Fe, Al, Ca, Sr, Ba, Ce, La, Zr, P with a 1.0 to 10.0 μm size, become effective graphite nucleation sites in commercial grey cast irons. The formation and activation of these compounds to be suitable as nuclei of graphite particles, at lower eutectic undercooling, usually require the following conditions for the final iron chemistry [6-12]:

- (%Mn) x (%S) = 0.03 - 0.06
- Al and / or Zr = 0.005 - 0.010%
- at least one of the inoculating elements Ca, Sr, Ba, Ce, La, usually up to 100ppm residual level in the iron.

The effects of inoculation are at a maximum after adding the inoculant and it has dispersed in the iron / dissolved, and the enhanced nucleation fades with time. The rate of fading depends on the inoculant composition, the type of iron to which it is added, and the iron temperature [13].

The objective of the present paper is to describe the fading characteristics of grey cast iron in the ten minutes after inoculation and the possibility to restore the preferred iron microstructure by re-inoculation after this 'holding' period.

It is worth noting that inoculation has become a series of additions in many foundry practices. Whereas inoculation used to be a single addition to a pour ladle, the adoption of autopour furnaces coupled with more demanding iron chill control has resulted in foundries making multiple inoculant additions between the furnace and the mould. This type of practice could also be referred to as re-inoculation. In these tests solidification rates represented in a wedge type casting were considered to evaluate the influence of different cooling rates on the fade effect and how they altered the microstructure of the test iron.

## 2. Experimental procedure

The test irons were melted in an electric induction crucible furnace (10kg, graphite crucible, 8000Hz), with the following thermal regime: superheating to 1550$^0$C and tapping at 1500$^0$C temperatures. A hypoeutectic cast iron with 3.72% carbon equivalent (CE), 3.35% C, 1.65% Si, 0.35% Mn, 0.03%S, 0.003%Al, 0.13% Cu, (%Mn) x (%S) = 0.011 chemical composition was selected for these tests.

An addition of 0.5% Ca-FeSi75 alloy was made as an in-stream inoculation during furnace tap. Inoculated irons were held for different times in the induction furnace before casting. After previously inoculated iron had been held for 11.5 minutes, it was re-inoculated using 0.3% Ca-FeSi75 as an in stream addition.

Wedge samples of the $W_{3½}$ type, ASTM A367 [25x40x120 mm size, with 0.45cm Cooling Modulus] [14] were cast in green sand moulds. A macroscopic analysis of the chill tendency was conducted on the wedge samples and the microstructural characteristics [graphite, carbides and matrix] were analysed. Fractures of the $W_{3½}$ samples were polished and analyzed metallographically, unetched, and etched with Nital (2%), along the geometrical centreline of the chill wedge at different distances from the apex of the wedge.

A controlled procedure was employed to record the degree of chill. Since immediate results were not required it was possible to avoid any hot shakeout effects on the solid state transformations, by deliberately delaying removal of the chill wedge castings from the mold, before analyzing the fracture.

Specific solidification conditions promote the occurrence of free carbides in a casting as clear chill (white iron area only) and / or total chill, which includes mottled iron, and these are usually measured to determine acceptability (Fig. 1) [15]. If chilled iron castings are being considered for wear resistance properties the transition zone from clear chill to total chill could also be important.

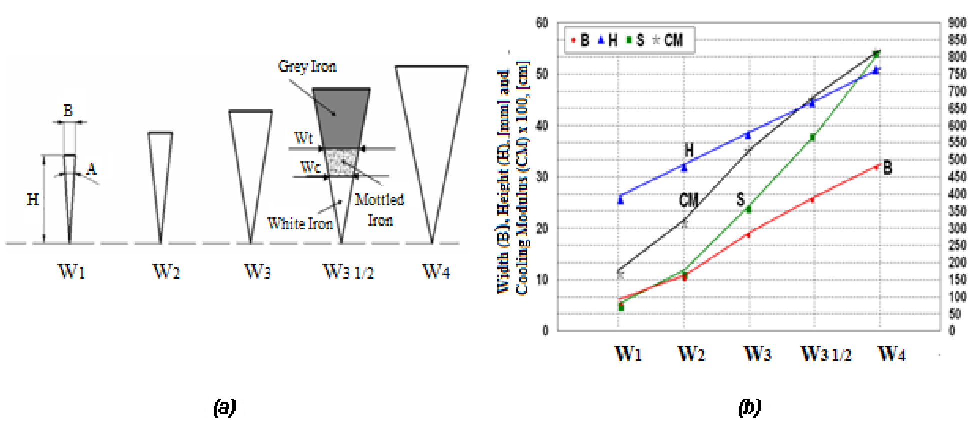


Fig. 1 Geometry of standard wedge shaped samples (a) and relationship of size parameters (b) [$W_c$ – clear chill; $W_t$ – total chill] [ASTM A367]

## 3. Results and discussion

### 3.1. Chill [Carbides] tendency

Both cooling rate and inoculation are important and related factors, but a number of trends in the chill tendency could also be identified. The iron solidification mechanism allows the formation of chilled iron microstructures, especially at lower carbon equivalent, in the hypo-eutectic iron range. This behaviour is aggravated either by increasing cooling rate or by decreasing the inoculation effect.

Graphitic cast irons are selected for their excellent machinability, whereas chilled microstructures detract from machinability, necessitating remedial heat treatment, and results in non-conformance with specifications, with increased total cost of production. The formation of carbides in cast iron, referred to as white iron or "chill" is quantified by measuring "chill width" as the white portion of a wedge fracture. The formation of iron carbides is more likely in thin sections with rapid cooling than in slower cooling, thicker sections.

Inoculated cast iron means actively changing the nature and chemistry of the particles, which make the precipitation of carbon as graphite easier.

Inoculation is a means of controlling the structure and properties of cast iron by minimizing undercooling and increasing the number of nucleation events during solidification; when inoculation is inadequate, and there are several reasons why, the resultant chilled structures are undesirable [5]. In the present experiments (Fig. 2, Fig. 3, Fig. 4), inoculation led to a marked increase in the

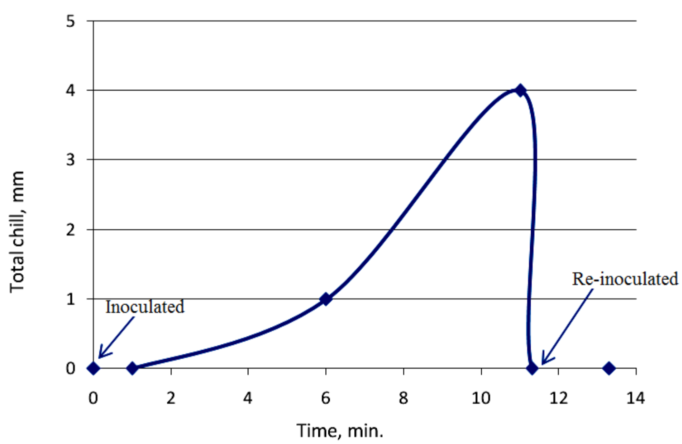


Fig.2 Influence of inoculation and holding time on chill tendency
[$W_{3\,½}$ wedge sample, ASTM A 367]

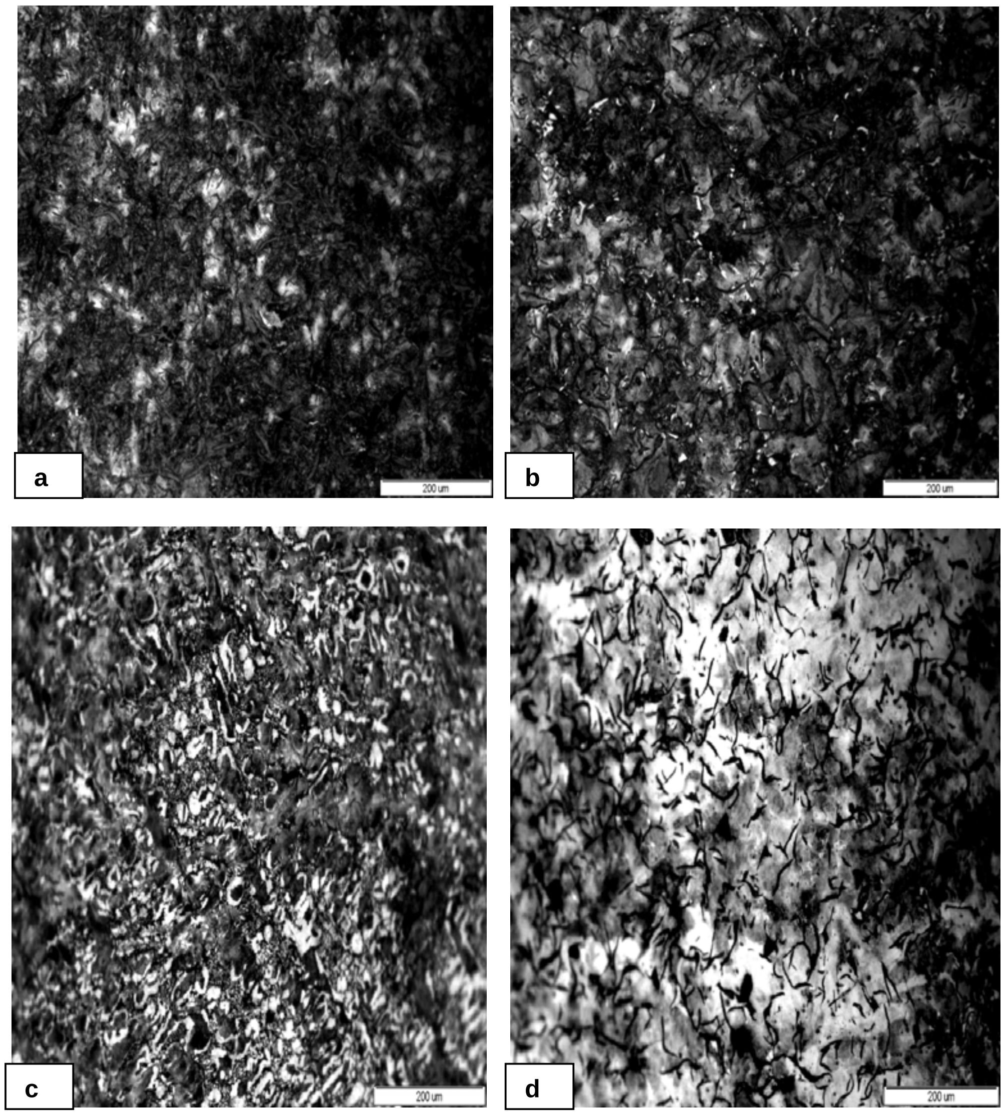


Fig. 3 Microstructure of cast irons at different hold time after inoculation [Nital 2%]; [a) – inoculated iron; b) - 5 min. hold; c) - 10 min. hold; d) - 11.5 min. hold and re-inoculation] [23mm from the apex, $W_{3\ 1/2}$ wedge sample, ASTM A 367]

number of suitable nucleation sites for graphite growth during eutectic solidification, thereby avoiding carbides formation, even at the highest solidification rates [the apex area of the wedge sample is the rapidly cooled section]. The effects of inoculation fade with time. The chill tendency increased

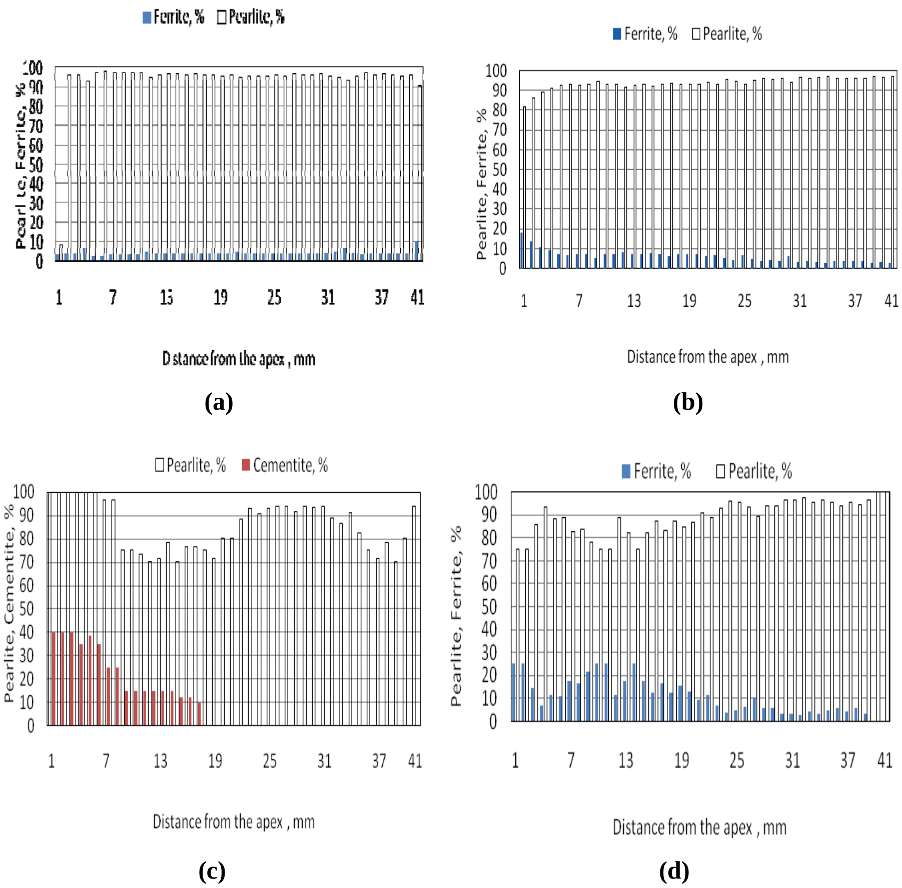


Fig. 4 Influence of inoculation and hold time after inoculation on metal matrix structure at different distances from the apex of $W_{31/2}$ wedge sample, ASTM A 367 [a) inoculated iron; b) 5 min. hold; c) 10 min. hold; d) 11.5 min. hold and Re-inoculation] [Pearlite + Ferrite = 100%]

with holding time, as seen at 6 min. after inoculation, and is four times higher after holding the iron 10 min.

The amount of cementite reached 40% of the surface area after 10 minutes holding time in the thinner sections of the wedge casting, typically up to 5 mm from the apex and was less than 20% at 18 mm from the apex; no carbides were found in the thicker sections during analysis of the wedge samples, either in the macro or micro examination (Fig. 3c, Fig. 4c).

Re-inoculation of the inoculated iron after holding for 10 minutes recreated favourable conditions for graphite nucleation, including the conditions with the highest solidification rate. No chill (carbides) was found in the re-inoculated iron, regardless of the cooling rate (Figs. 3d, 4d).

### 3.2. Graphite phase characteristics

Mn, S and Al contents using the (%Mn) x (%S) parameter (0.011), were lower than the recommended range 0.03–0.06, with less than 0.004% Al, compared to a preferred range 0.005–0.01% Al. The contents of Mn, S and Al were intentionally chosen in these critical ranges, along with a higher bath temperature, to develop an iron that would be more likely to solidify with an undesirable microstructure. Usually, these irons solidify at higher eutectic undercooling, typically at $\Delta T_1 < 20^oC$ (less than recommended more than $25^oC$). This is typical of grey irons electric melted in the new generation of acid lined, medium frequency coreless induction furnaces.

Graphite type, size and shape formed during cast iron solidification, as well as the amount of graphite versus iron carbide, can be controlled by inoculation. Amount (% surface) and size (particle length) of the lamellar graphite morphology are visibly dependent on the solidification conditions, inoculation and holding time after inoculation, respectively (Fig. 5).

Inoculated cast iron is characterized not only by the absence of the carbide phase, but also by different graphite parameters, strongly influenced by the cooling rate. For the wedge type casting solidification, the cooling rate varies not only under the influence of the section thickness, but is also subject to an "end effect" (high cooling rate, despite the highest section thickness). The corners of the widest part of any wedge behave as thin sections.

In inoculated iron, undercooled graphite morphologies, which typically appear as small particles (less than 20-30 μm), are present only at the highest cooling rates as in thin wall sections (up to 3 mm from the wedge apex) or at the "end effect". With an extended holding time after inoculation, the graphite phase characteristics deteriorate with less of the type A graphite morphology in the microstructure.

After holding the inoculated iron for 5 minutes the typical type A graphite is only present in the thicker section of the wedge (furthest from the apex). After iron is held for 10 minutes this graphite morphology practically disappeared and a fine sized undercooled graphite formed independent of the cooling rate throughout the wedge sample.

Re-inoculation after a 10 minutes holding time restored more favourable graphite nucleation conditions, and consequently, a smaller sized (40-100 μm) type A graphite, with a more uniform distribution, was formed. It appears that this treatment is efficient for critical melting and solidification conditions, including un-favourable chemistry of the base iron (Mn, S and Al content), superheating in the melting furnace (typically for induction furnaces) and high cooling rate solidification (typically for thin wall castings).

### 3.3. Base metal matrix characteristics

Ferrite and pearlite are the most important components of the base iron matrix, as cast [Ferrite + Pearlite = 100%]. For the common iron compositions the Pearlite / Ferrite ratio is determined not only by the solidification (cooling rate) but also by graphite phase characteristics, which control carbon diffusion during the eutectoid reaction.

In these test conditions the inoculated iron had a matrix with 90-95% pearlite and 5-10% ferrite, typical of a hypoeutectic grey cast iron at 3.72% Carbon Equivalent (CE). A relatively uniform distribution of the graphite phase

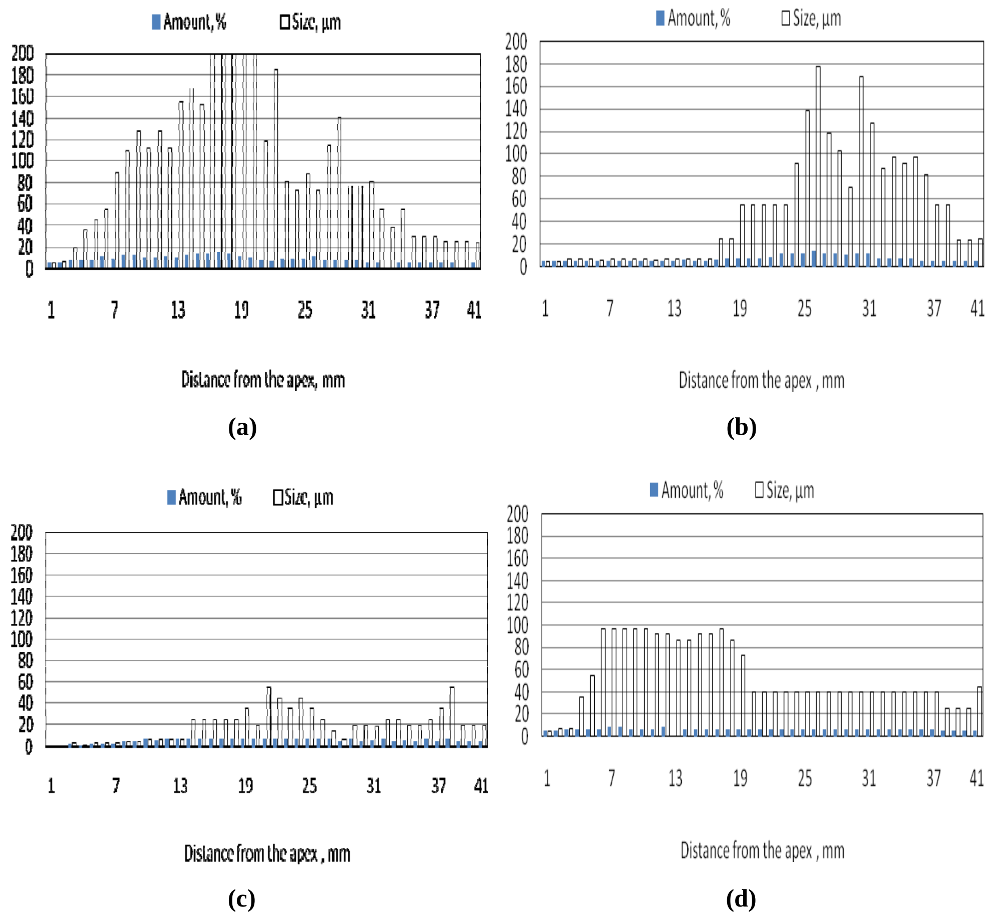


Fig. 5 Influence of inoculation and hold time after inoculation on graphite phase parameters at different distances from the apex of $W_{31/2}$ wedge sample, ASTM A 367 [a) inoculated iron; b) 5 min. hold; c) 10 min. hold; d) 11.5 min. hold and Re-inoculation]

characteristics (morphology, amount and size) does not affect the pearlite / ferrite ratio in a specific section of a wedge type casting.

With increasing hold time after inoculation the pearlite / ferrite ratio was affected, favouring pearlite formation, when the structure solidified white (non-graphitic) or favouring ferrite formation, when the structure contained fine sized undercooled graphite. The pearlite / ferrite ratio is less influenced 5 minutes after inoculation, than the more visible effect after 10 minutes, especially as ferrite formation in the areas containing undercooled graphite. Re-inoculation restored a relative uniform distribution of pearlite / ferrite ratio, but with a slightly higher ferrite presence: 75-90% pearlite and 10-25% ferrite.

## 4. Conclusions

*It was confirmed that inoculation fade might be very rapid with much of the effect lost within the first few minutes after inoculation. To preserve the maximum effect, the inoculated iron must be cast soon after inoculation with a target pour time of 5 minutes;

*Holding inoculated molten iron increases undercooling during eutectic solidification, with an increased tendency to chill formation, especially in all rapid solidification rate areas, not just thin section but also where there is an "end effect" or "corner effect";

*Severe fade of the inoculation effect can also promote the formation of ferrite associated with undercooled graphite in grey cast irons;

*A second small addition of inoculant, in a late inoculation practice is recommended to restore most of the typical inoculation benefits;

*It was found that the re-inoculation of grey cast irons reduces the tendency to chill and mottle, so avoiding embrittling carbides and reducing the risk of cracking during shake-out or fettling, and the need for remedial heat treatment, inclusively for critical solidification conditions, typically for electric melting irons [(%Mn) x (%S) < 0.03; Al < 0.005%].

*If late inoculation is already conducted, conventional foundry practice would be to limit the time that any batch of grey iron is held after any prior (transfer type) inoculation, to avoid undesirable microstructures.

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