INOCULATION OF GREY AND DUCTILE IRON

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ABSTRACT

The demand of sophisticated castings is increasing and the foundries must follow this trend. That is why the supplier of Inoculants has a more and more important role in order to provide materials that can enable the production of the castings in terms of soundness, mechanical properties and machinability.

The aim of this paper is to review some important facts related to cast iron inoculation. The mechanism of inoculation and graphite nucleation in grey and ductile irons are explained. Finally, the effects of elements such as Ca, Ba, Sr, Bi, Ce on cast iron solidification will be shown. Last but not least theoretical aspects & practical experiences are described in detail in order to underline how important efficient inoculation for the soundness of castings is.

Keywords: Cast iron, inoculation, graphite nucleation, inoculants
1- INTRODUCTION

The inoculation of liquid cast irons in order to control the final structure and thus mechanical properties has been deliberately practiced for more than 100 years.

By the 1920’s and 1930’s the inoculation of grey irons with Ferrosilicon and Calcium Silicon was commonplace.

In the 1940’s the recognition and understanding of inoculation and inoculants had advanced considerably and “special Inoculants” became commercially available (see table 1 & 2). The better performance of these Inoculants, compared to standard Ferrosilicon, was confirmed by the Foundries.

Today, the foundryman is “spoiled for choice” with the wide variety of inoculants available to him from several producers. In this paper, we shall be frequently discussing inoculants both FerroPem products and those of our competitors. As a background to these discussions, it is useful to briefly review the importance of inoculation, as well as some of the proposed explanations for how inoculants work (the mechanism of inoculation) and the importance of, chemical composition – the role of specific individual, and combinations of, elements employed in the formulation of inoculants.

Table 1: Typical analysis of some ladle Inoculants

<table>
<thead>
<tr>
<th>No.</th>
<th>INOCULANT</th>
<th>C (%)</th>
<th>Ca</th>
<th>Cr</th>
<th>Mn</th>
<th>Si</th>
<th>Ti</th>
<th>Zr</th>
<th>Al</th>
<th>Fe</th>
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<td>1.</td>
<td>Ca-Metal</td>
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<td>100</td>
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<td>2.</td>
<td>Ca-Si</td>
<td>30-35</td>
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<td>3.</td>
<td>Ca-Si-Ti</td>
<td>5-8</td>
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<tr>
<td>4.</td>
<td>Cr-Si-Mn-Ti-Ca</td>
<td>3.0</td>
<td></td>
<td>38.42</td>
<td>8.11</td>
<td>14.16</td>
<td>1.50</td>
<td>bal.</td>
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<tr>
<td>5.</td>
<td>Cr-Si-Mn-Ti-Ca</td>
<td>3.0</td>
<td></td>
<td>28.32</td>
<td>14.16</td>
<td>15.21</td>
<td>1.50</td>
<td>bal.</td>
<td></td>
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<td>6.</td>
<td>Cr-Si-Mn-Zr (3 grades)</td>
<td>30-52</td>
<td>5-10</td>
<td></td>
<td></td>
<td>14.35</td>
<td>1.6</td>
<td>bal.</td>
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<tr>
<td>7.</td>
<td>FeSi</td>
<td>0.5-0.8</td>
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<td>8.</td>
<td>FeSi</td>
<td>0.5-0.8</td>
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<td>9.</td>
<td>Graphite</td>
<td>90-100</td>
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<td>10.</td>
<td>Mo-Si**</td>
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<tr>
<td>11.</td>
<td>Ni-Si***</td>
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<td>12.</td>
<td>Si-C</td>
<td>28-46</td>
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<td>13.</td>
<td>Si-C</td>
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<td>14.</td>
<td>Si-Mn</td>
<td>2.5</td>
<td></td>
<td>20-25</td>
<td>5-7</td>
<td>60-65</td>
<td>5-7</td>
<td>1.75</td>
<td>bal.</td>
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<td>15.</td>
<td>Si-Mn-Zr</td>
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<td></td>
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<tr>
<td>16.</td>
<td>Si-Ti</td>
<td></td>
<td></td>
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<tr>
<td>17.</td>
<td>Si-Zr</td>
<td></td>
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<tr>
<td>18.</td>
<td>Si-Zr</td>
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* Approximate
** 60% Mo
*** 60% Ni
Table 2: Typical analysis of some ladle Inoculants

<table>
<thead>
<tr>
<th>Type of Inoculant</th>
<th>C%</th>
<th>Si%</th>
<th>Al%</th>
<th>Ca%</th>
<th>Ba%</th>
<th>Sr%</th>
<th>Zr%</th>
<th>Mn%</th>
<th>Ti%</th>
<th>FeSi</th>
<th>FeSi - Ba</th>
<th>FeSi - Al</th>
<th>FeSi - Ca - Ba</th>
<th>FeSi - Mn</th>
<th>FeSi - Sr</th>
<th>FeSi - Ti</th>
<th>FeSi - Fe</th>
<th>FeSi - FeSi</th>
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<tr>
<td>FeSi standard</td>
<td>45-50</td>
<td>30-33</td>
<td>0.6-0.8</td>
<td>0.3-0.7</td>
<td>4-6</td>
<td>0.4-0.7</td>
<td>0.8-1.3</td>
<td>8-10</td>
<td>5.5-6.5</td>
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<td>FeSi - Ba - Ca</td>
<td>72-78</td>
<td>30-33</td>
<td>0.6-0.8</td>
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<td>0.4-0.7</td>
<td>0.8-1.3</td>
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<tr>
<td>FeSi - Al - Ca - Ba</td>
<td>64-68</td>
<td>0.6-1.1</td>
<td>1.4-2</td>
<td>0.6-1</td>
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<td>0.4-1.7</td>
<td>5.5-6.5</td>
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<tr>
<td>FeSi - Mn - Ba</td>
<td>60-65</td>
<td>0.6-1.1</td>
<td>1.4-2</td>
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<td>0.4-1.7</td>
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<tr>
<td>FeSi - Sr - Ti</td>
<td>63-67</td>
<td>0.2-0.5</td>
<td>&lt;0.1</td>
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<tr>
<td>FeSi - Mn - Sr</td>
<td>70-75</td>
<td>0.2-0.5</td>
<td>&lt;0.1</td>
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<td>FeSi - Fe - Fe</td>
<td>75-80</td>
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</tbody>
</table>

*FeSi = FerroSilicon; d = determined; Si = Silicon; Mn = Manganese; Ti = Titanium*
2- WHAT IS INOCULATION

The main aim of inoculation is to promote the solidification according to the stable system iron-carbon. This is done by preventing undercooling below the metastable eutectic temperature where iron carbides (Fe3C) are formed.

The inoculation of grey and ductile iron is realized by the addition of small amounts of materials known as inoculants. An inoculant is a ferro-silicon based alloy added to the molten metal either just before or during the pouring of the liquid iron into the mould.

The inoculant will provide sufficient nucleation sites for the dissolved carbon to precipitate as graphite rather than iron carbides (cementite).

Inoculants can be found in several sizes which depend on the foundry process. Indeed the inoculant addition can be made in the furnace, in the ladle, in stream or in the mould.

Additionally, inoculation improves mechanical properties (strength and ductility) of the Iron.

Figure 1: Eutectic cells in Grey Cast iron
3- WHY INOCULATE CAST IRON

The cast iron mechanical properties and final structure can be influenced either during the solidification or during a heat treatment. In order to optimize the cost of production, the required structure should be achieved during solidification as-cast.

However, according to Fe-C diagram, carbon can solidify under two forms, either graphite or Fe3C cementite. This corresponds to the two phases diagram:

1) \( L \rightarrow Y + C \) **STABLE**  
   (liquid)  (Austenite)  (graphite)

2) \( L \rightarrow Y + \text{Fe}3\text{C} \) **METAStABLE**  
   (liquid)  (Austenite)  (Iron carbide)

The simplified equilibrium diagrams showing these possible eutectic transformations are shown in Fig 2 & 3.

During the solidification, the cast iron reaches the eutectic temperature. At that moment, it should solidify according to the stable Iron-Graphite diagram. However equilibrium conditions never occur in practice and there is always a degree of undercooling followed by recalescence before solidification is completed as shown in Fig.4 next page.
The implications of this undercooling phenomenon in the solidification of cast irons (grey and ductile) are clearly seen in Fig. 5 & 5bis.

**Figure 4: Undercooling phenomenon**

**Figure 5: Formation of Grey, White and Mottled irons**

**Figure 5bis: Formation of Grey, Vermicular and Ductile irons**
In most grey and ductile irons, the presence of the metastable eutectic carbides (chill) is undesirable.

Therefore the main reason for inoculation is to provide nuclei in the liquid iron melt, which enhances the graphite nucleation with a low degree of undercooling and therefore the solidification will follow the stable Iron-Graphite Diagram.

This will avoid the formation of iron carbides (chill) and consequently results in good mechanical properties.

The effect of inoculation in reducing the degree of undercooling is shown in Fig 6.

![Figure 6: Cooling curves for inoculated and uninoculated irons](image)

**The two most important factors which influence the formation of carbides are:**

1 – The number of **stable nuclei** in the liquid which can act as sites for the growth of the eutectic graphite.

![Figure 7: Barium Oxysulphide](image)

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2 – The **cooling conditions**, which are influenced by the wall thickness and geometry of the casting.
All liquid irons, when tapped from a melting furnace (cupola, electric induction, rotary etc…), have an inherent level of nuclei. This level depends on the method of melting, type of charge materials used, temperature and so on.

![Figure 8: Structures of ductile irons before and after inoculation](image)

The main effects of inoculation for both Grey and Ductile Iron are described as hereunder:

**Grey cast iron:**

- reduces chill and promotes graphite formation
- reduces the formation of fine graphite
- correct Inoculation leads to the desired Graphite size
- promotes uniform structures in various sections
- improves mechanical properties
- improves machinability

![Figure 9: Structures of poorly and well inoculated grey irons](image)
**Ductile iron:**

- reduces tendency for chill
- increases the nodule count
- promotes the formation of fully spheroidal graphite
- Ferrite structure as-cast in thin sections possible

![Figure 10: Structures of poorly and well inoculated Ductile irons](image)

4- **INOCULATION MATERIALS**

**Carbon (graphite)**

In order to promote graphite during solidification of liquid cast iron, an obvious choice would be carbon. However, only crystalline graphite, such as naturally occurring graphite or crushed graphite electrode scrap, is an effective inoculant. Amorphous forms of carbon such as metallurgical coke and carbon electrode scrap are not suitable inoculants.

Crystalline graphite is rarely used alone as an inoculant but is usually mixed with ferrosilicon based inoculant such as the FerroPEM inoculant INOCARB™.

INOCARB™ is a mixture of 47 % graphite with Ba-based ferrosilicon inoculant.

INOCARB™ is extremely effective in reducing chill of thin/medium section in Grey Iron Castings. Additionally the lower level of aluminium is useful in avoiding pinhole defects in grey iron.

Inoculants based on graphite (including INOCARB™) are generally not suitable for ductile iron.
Ferrosilicon based inoculant

Pure silicon and pure Ferrosilicon alloys are not very efficient as ladle inoculant.

Indeed, silicon based inoculants require to have at least some elements such as Aluminium, Calcium, Barium, Strontium, Zirconium, Cerium and Bismuth to produce sufficient stable nuclei therefore the inoculation effect.

The most commonly used silicon based inoculant is 75 % Silicon ferrosilicon, which normally contains between 1 and 2 % Aluminium and 0.2 % and 1 % Calcium.

The use of ferrosilicon in the 1930’s and 1940’s gave variable results since the levels of Aluminium and Calcium were inconsistent. Even today, many commercially available 75 % Silicon ferrosilicon inoculants have variable levels of Aluminium and Calcium (they are present as impurities in the raw materials used to produce the ferrosilicon) and consequently, their inoculation effect is erratic.

The specially formulated inoculants are all generally more efficient than normal 75 % Silicon ferrosilicon.

Calcium has long been recognised as an important component of inoculants.

Calcium is a very strong deoxidiser, considered by many to be part of the mechanism of inoculation.
Barium functions similar to Calcium (strong deoxidiser) and contributes to a strong nucleation effect.

However, Barium seems to give a better efficiency to the inoculant over a wider temperature range than Calcium alone. Tests are reported showing that Barium is efficient as a nucleating agent, when added to liquid cast iron at temperatures in excess of 1480°C. However at temperatures ranging from 1370°C to 1430°C, Calcium was found to be more effective than Barium.

Thus the combination of Barium and Calcium in an inoculant such as INOBAR™ (9% Ba; 1% Ca) makes it effective over a wider range of temperature and, additionally, the Barium appears to significantly reduce fading and thereby increases the fading time after inoculation.

The FerroPEM inoculant GRAFIDIN™ is a well-conceived formulation with levels of Ca and Ba at 2.5 and 4.5 % respectively and a Manganese level at 9 %. Indeed the effect of Manganese in a ferrosilicon based inoculant is to reduce its melting point thus making its dissolution in the liquid iron more rapid even at relatively low temperatures (1330 – 1350°C).

The FerroPEM inoculant LMC™ or Inocast 175 contain lower levels of Ca and Ba in addition to Aluminium. They are universally applicable inoculant of both grey and ductile iron.
Inobar is often used as a pre-conditioning agent for Grey and Ductile Iron or as an inoculant for heavier sections due to its good antifading properties.

Inobar contains typically 9% Ba. As an Alkaline-earth, it forms Oxysulphides of barium with Oxygen and Sulphur (BaSO4) which provide a high number of stable nuclei.

The study below (fig.13) from JC.Percheron (PEM) shows, that at 9% barium, we can reach a remarkable reduction of Chill. Over 9% the chill reduction is at a stable level and isn’t likely to be reduced.

The graph below (fig.14) (author: Morgan), compare the efficiency of different inoculants over time.

A Strontium (curve 7) based inoculant promotes a high nodule count just after inoculation and its efficiency decreases over time. Whereas Barium based inoculant (curve 5) maintain a constant and good efficiency over time.

According to the graph below the fading time of Inobar is stable until 20 min, with the highest nodule count of all tested inoculants.
Zirconium has a chemical affinity with several impurities found in liquid iron melts.

It is a deoxidising element with the ability to react with nitrogen and sulphur. Zirconium is an important component of Ferropem’s inoculants such as ZIRCOGRAF™ and ZL80™…

ZIRCOGRAF™ contains high levels of Zirconium (6 %) and Manganese (6 %) in addition to the important contents of Calcium and Aluminium.

The high level of Manganese reduces the melting point of ZIRCOGRAF™, which makes it particularly useful for relatively low metal temperatures and also for late-stream inoculation, where quick dissolution is required.

ZL 80™ is another complex ferrosilicon multi-element inoculant containing Al (1.4 %), Ca (2.6 %) and Zr (1.6 %). ZL 80™ is universally recognized as a powerful and consistent inoculant which is suitable for use with both Grey and Ductile iron. Its strong chill-reducing power, coupled with its slow fading characteristics have been confirmed during more than 20 years of use by foundries.
All the elements discussed previously make their own contribution to improve the efficiency of Ferro Silicon based inoculants. They all assist in reducing chill by increasing the number of eutectic cells. However, in grey iron an excessive number of eutectic cells can cause a reduced Graphite size.

The use of Strontium containing inoculants can be of great benefit in grey iron, since excellent chill reduction can be achieved, although there is no increase in the number of eutectic cells.

To achieve this effect with Strontium, the level of Calcium must be kept below 0.1 % and the Aluminium below 0.5 %. Indeed it appears to be some interferences with strontium in the presence of Ca and Al in excess of these stated levels.

INOSTRONG™ contains the required amount of Al and Ca combined with Sr (0.9%). This low Al contents is a positive advantage in grey iron situations where H₂ pinholing may be a problem.

This inoculant is recommended for Grey Iron. Indeed, for the same degree of chill reduction, the eutectic cell number is lower. Consequently the tendency for shrinkage defects in grey iron is reduced.

INOSTRONG™ is also recommended for ductile irons without Ce in the process to avoid possible interaction between the Ce (RE) and the Sr.
Bismuth is a very powerful element promoting a high nodule count in ductile iron, as shown in Fig. 15.

Figure 15: Effect of Bismuth on nodule number and fading in ductile irons inoculated with ferrosilicon

The formulation of the FerroPEM inoculant SPHERIX™ includes 1% Bi and 0.5 % RE. This inoculant is described later in the paper.
Cerium

The presence of Cerium in ductile iron significantly alters the inoculating effects and fading characteristics of many inoculants. For most inoculants, the presence of Cerium increases nodule count, decreases the chill tendency and reduces the fading-time.

In addition to the increased nodule count and better fading characteristics, CERINOC™ is also useful in case of cast surface degeneration of graphite (flake graphite skin) due to metal mold reactions.

Figure 16: Effect of Cerium on nodule number and fading in ductile irons inoculated with ferrosilicon

Lanthanum

FeSiLa is an inoculant for Ductile and Vermicular Irons specifically alloyed with Lanthanum.

It is an efficient inoculant for chill reduction, but the main aim of this inoculant is to reduce shrinkage tendency in Ductile Iron.

It has been proved that Lanthanum promotes the Equiaxe solidification instead of the classical columnar solidification. Thus during the solidification, particles of Iron can grow freely in the liquid, in all directions and improve the self-feeding of the casting.
Experimental work

An iron melt, was treated with different quantities of La (0.15 % of inoculant containing from 0 to 2 %La). Then the treated melts were poured at the same temperature in a cylindrical mold.

The objective of this trial was to compare the thickness of solidificated Iron in the mold with and without La.

The moulds have been emptied after the same period of time (about 50 % of the solidification was reached). From both samples the wall thickness was measured. The use of La results in thinner wall, which means less tendency for columnar solidification. Consequently the gating system and critical parts of the casting remain longer “open” and are able to feed.

The two pictures below show the result:

Without La
Microshrinkage

Without FeSiLa
No Microshrinkage

Without La
Thickness 6 mm

With La
Thickness 2 mm
Spherix is based on 75% Silicon and contains strictly controlled proportions of Bismuth and Rare Earths (RE) with a ratio of 2:1. It combines the beneficial effects of both Bi and RE in order to improve nucleation performance by increasing the Nodule Count and reducing the fading. The use of Spherix results in a reduction of chill, particularly in fast cooling thin section castings. Thus a ferritic as-cast structure can be achieved, which is desirable in order to keep the production cost under control.

Bismuth should never be added to ductile iron unless Cerium is also present. Without Cerium, for low amounts (20 ppm in the Iron) Bismuth produces undesirable graphite forms. There are some foundries using Bi metal in their process. This is risky, as the use of a pure metal, added to the melt in very small amounts can cause problems of homogenous distribution in the liquid iron. In addition Bi is a highly volatile element at high temperatures as the vapour pressure is very high. Very often a yellow smoke can be seen, when Bi-metal is added in the melt.

Consequently BiRE in a Ferrosilicon alloy is easy to use and precise, as the ratio Bi/RE, which is important for the metallurgical result, is always respected.

Spherix is beneficial in the following cases:

- For base Iron with a high level of trace elements,
- For Iron treated with pure Mg (S and O low)
- For sophisticated casting designs
- Inoculation process optimisation (reduction in addition rate)
- To avoid metallurgical defects such as chunky, chill, inverse chill…
- To improve machinability
- To increase the ability to produce more complicated castings (competitiveness advantage)

**Case study with Spherix**

This trial has been carried out in an automotive foundry. The objective of the trial was to improve the mechanical properties of the casting (clutch plate with thin sections).

The standard process used was the following:

1. Nodularisation process with pure Mg (Converter)
2. Ladle inoculation with 0.4% Standard FeSi
3. Late inoculation 0.1% Ba based inoculant

For the industrial trial, the late inoculant was replaced by Spherix. A step wedge from 5 to 40 mm was analyzed to compare the results of both processes:

- The reference process
- The Bi/RE Inoculant
Influence of Bi/RE inoculant on Nodule Count

The figure 17 below shows the Nodule count for the reference and Bi/RE inoculant process on a step wedge from 5 to 40 mm.

The trial was made for 0.1% and 0.2% addition rate of inoculant.

With the Bi/RE inoculant, the nodule count is higher and independent from thickness.

![Figure 17: Comparison of nodule count](image)

Influence of Bi/RE inoculant on pearlite

The figure 17 below shows the pearlite level for the reference and Bi/RE inoculant for a step wedge from 5 to 40 mm.

The trial was made for 0.1% and 0.2% inoculant addition rate.

With the Bi/RE inoculant, the pearlite rate is more constant. Besides, the Bi/RE inoculant process is able to reduce pearlite levels and so increases ferrite levels as-cast.

![Figure 18: % pearlite](image)
5- CONCLUSION

1. Although the amounts of inoculants used are low, inoculation is an important step in the production of Grey and Ductile Iron Castings.

2. The choice of the inoculant depends on the following factors:
   - Charge materials
   - Melting technology
   - Mag treatment
   - Moulding technology
   - Type and section thickness of the castings …
   - Specific customer requirements
   - And so on

3. Speciality inoculants, which are the FerroPEM range of products, have been developed for various specific applications. The chemical composition of each of the inoculants has been carefully developed thanks to the knowledge of the individual and combined customer demands and Ferropem’s R&D competence.

4. High quality standards in our plants result in good and consistent products, that can be designed also“custom made” for specific customer requirements.

5. Inoculants are complex Ferrosilicon based products with various elements like Bi, Sr, Zr etc., which have a non deniable contribution to the soundness and desired structure of the casting.

6. The selection of the grain size of the inoculant is also a very important factor influencing the dissolution and thus efficiency of the Inoculant.
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