INVESTIGATION OF POSTINOCULATION PARAMETERS OF SPHEROIDAL GRAPHITE IRON PRODUCTION

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Presented by
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Approval of the Graduate School of Natural and Applied Sciences.

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ABSTRACT

INVESTIGATION OF POSTINOCULATION PARAMETERS OF SPHEROIDAL GRAPHITE IRON PRODUCTION

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M.S. in Metallurgical Engineering
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In this study, several experiments were performed aimed at postinoculation of spheroidal graphite iron, which has a large application fields at industry.

The study consists of two main parts. The first part is investigation of postinoculation parameters of spheroidal graphite iron, and second part is the finding out of time effects on microstructure.

The samples, which were poured to sand molds, were investigated using metallographical methods.

As a result, parameters of postinoculation of spheroidal graphite iron production were determined. Optimum times of postinoculation are established.

Key words: Postinoculation, Inoculation, Spheroidization

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ÖZ

KÜRESEL GRAFİTLİ DÖKME DEMİR ÜRETİMİNDE SON Fe-Si AŞILAMASININ DEĞİŞKENLERİ

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Bu çalışmada, endüstride uygulama sahası çok geniş olan küresel grafitli dökme demirin üretiminde kullanılan son Fe-Si aşılamasında etkili olan değişkenler araştırılmış ve zaman değişkeninin mikro yapıya etkileri ortaya çıkarılmıştır.

Numuneler, kum kalıba dökülmüş ve metalografik metodlar kullanılarak incelenmiştir.

Sonuç olarak, küresel grafitli dökme demir üretimindeki son Fe-Si aşılamasının değişkenleri belirlenmiş ve son Fe-Si aşılamasındaki optimum zamanlamalar tespit edilmiştir.

Anahtar Kelimeler: Son Fe-Si aşılaması, Aşılama, Küreselleştirme.

Bilim Dalı Sayısal Kodu: 604.02.04

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TABLE OF CONTENTS

LIST OF TABLES

LIST OF FIGURES

CHAPTER I

INTRODUCTION

Spheroidal graphite iron (S.G.I) is the last member of cast iron family. It has most of the advantages of gray iron and steel. Good wear resistance, excellent machinability, low melting point,good fluidity and castability are similar advantages of S.G.I and gray iron, also high strength,toughness, ductility, hot workability and hardenability are similar characteristics of steel and S.G.I.

S.G.I has got many applications at industry. Automotive and diesel-cranksafts, pistons and cylinderheads; mining-hoist drums, drive pulleys, flywheels, and elevator buckets; electrical fittings, switch boxes, motor frames, and circuit-breaker parts ; agricultural-tractor and implement parts; steel mill-work rolls, furnace doors, table rolls, and bearings are some of the typical applications of S.G.I.

S.G.I which is also called by some other names as nodular cast iron, ductile iron and spherulitic iron, contains 3.0-4.0 %C, 1.8-2.8 %Si ,0.15-0.90 %Mn, and minor quantities of S and P together with of other trace elements.[1]

The structure of cast iron can be modified by small quantities of magnesium known as "Magnesium Inoculation" to the liquid metal just before casting operation. Magnesium inoculation causes the transformation of lamellar graphite to spherical shape, and also an additional inoculation by Fe-Si ensure well-shaped spheroidal and more nodule. This second Fe-Si inoculation, that is made after Mg inoculation, is known as postinoculation.[2]

Postinoculation of S.G.I, although used in many foundries, is far from being a standard procedure. In foundries using postinoculation major differences exist in amount of postinoculant, type and size of alloy, time of postinoculation and method of addition.

During the studies, it was realised that magnesium inoculation and postinoculation have great influence on microstructure and mechanical characteristics. In this study, postinoculation parameters of spheroidal graphite iron were investigated and time effects on microstructure were studied.

CHAPTER II

PRODUCTION OF S.G.I

2.1 Description of S.G.I

Chemical composition of S.G.I. can be defined as follow ;

C	3.0-4.0%
Si	1.8-2.8%
Mn	0.15-0.90%
P	0.1% max
S	0.02% max

If an iron alloy exceeds about 2% carbon, carbon does not have to nucleated from decomposition of austenite; it can form directly from the melt by an eutectic reaction. Cementite (Fe_3C) nucleate at the eutectic more readily than graphite, but on sufficiently slow cooling graphite itself is able to form and grow.

Cooling rate of cast irons can be investigated at 3 speeds.

1. rapid cooling

2. slow cooling

3. very slow cooling

1. Rapid cooling: As the alloy cools below the liquidus, dendrites of austenite form and grow until the eutectic temperature is reached. At the eutectic, graphite formation is suppressed, but austenite and cementite(Fe_3C) precipitate to form ledeburite, a form of eutectic which consists of spheres of austenite embedded in cementite. Ledeburite forms at Fe-Fe_3C eutectic, solid line (nm). On further cooling, the cementite grows as the

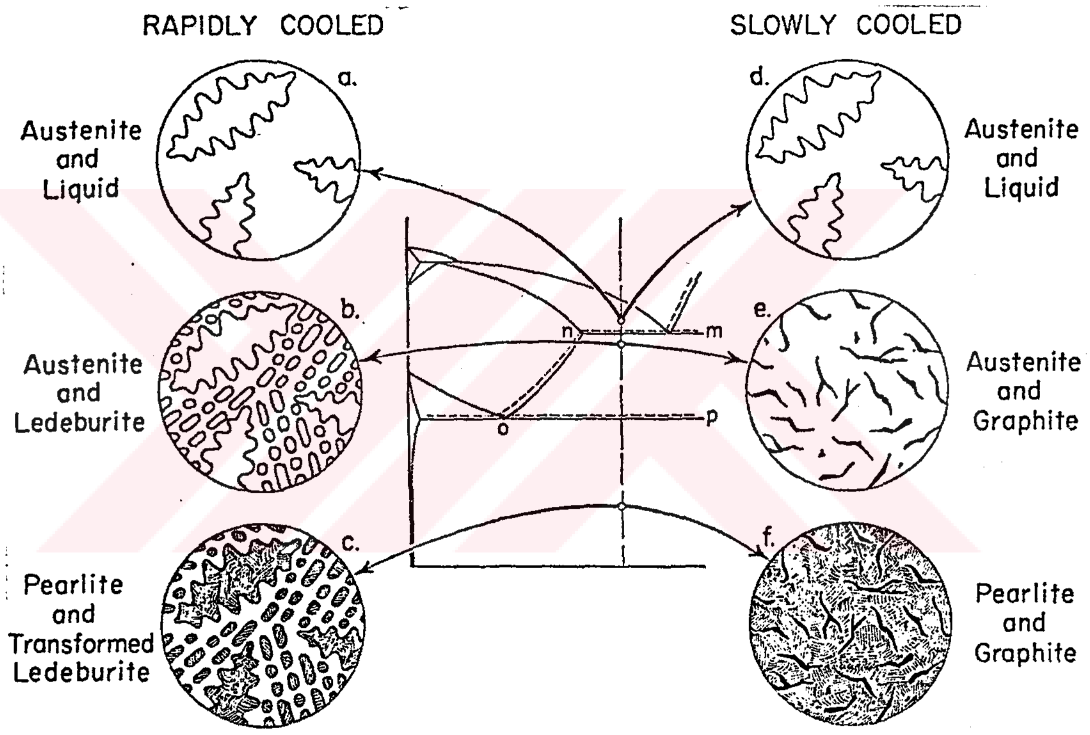


Figure 1. Microstructure obtained on solidification of cast iron. [1]

austenite decreases in carbon content along the solid line (no). At (o) the eutectoid reaction transforms the remaining austenite to pearlite. Microstructures illustrating the solidification and cooling of this alloy are shown in Figures 1 a, b, c. At the room temperature it is hard and brittle,and is called white iron because the surface of a fractured piece of the iron is white. [1]

2. Slow cooling: In this case, austenite first forms from the melt, but eutectic freezing is now slow enough so that the products of the eutectic reaction are austenite and graphite. The reaction takes place at the dotted line (no), and the graphite flakes grow. At the eutectic temperature (op), remaining austenite transforms as below to pearlite. Figure 1 d,e, and f show typical microstructures of an alloy cooled in this manner; the fracture surface appears dull gray, and the material is known as gray cast iron (or pearlitic gray iron).[1]

3.Very slow cooling: Phase changes in such an alloy will be exactly like those of case 2 above, except that, at the eutectoid, cooling will be sufficiently slow to permit graphite to precipitate rather than pearlite. Actually, no new graphite flakes will form, but ones present will increase in size. In this case final microstructure will be only graphite flakes embedded in a matrix of ferrite; this is termed ferritic gray iron. Sometimes the cooling rate of a portion of a casting may be intermediate between rapid cooling and slow cooling ; then a structure containing patches of both white iron and gray iron is obtained this is known as mottled iron.[1]

Graphite may be formed in cast irons as flakes, nodules, or spheroids (Figure 2).

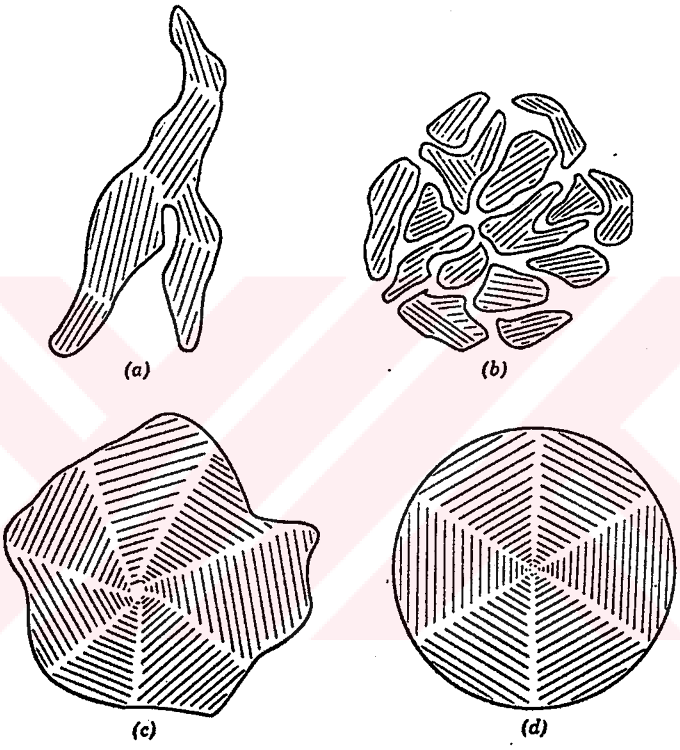


Figure 2. Forms of graphite in cast irons [1].

Regardless of shape, graphite has very little cohesive strength and reduces the effective metallic cross section of the casting. Graphite reduces tensile strength and ductility most in flake form; the flakes are often interconnected, and their relatively sharp edges are potential areas of low ductility and weakness. The graphite nodule is more favourable form. The ideal graphite shape from the viewpoint of strength and ductility is shperoidal.

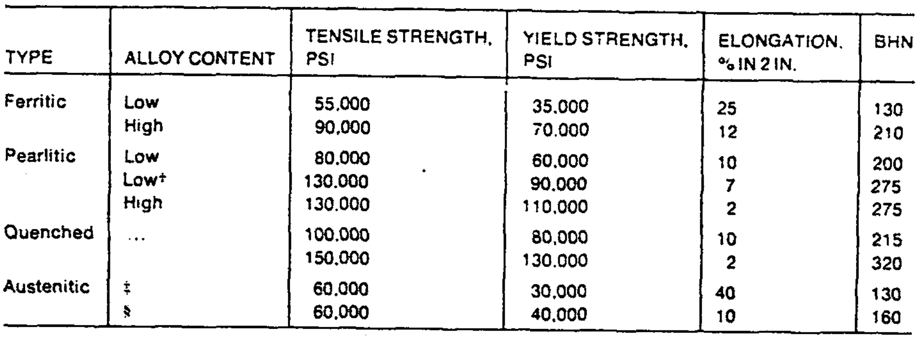
The importance of developing an adequate nodule count to produce high quality spheroidal graphite iron is very clear. The role of nodule count in controlling the amount of nonspheroidal graphite and primary carbides is also considered. Based on the fact that the control of the structures in S.G.I is depend upon the development of a sufficient nodule count and metal chemistry. Nodule count depends on 1)Carbon Equivalent, 2) Section size of the casting, 3) Magnesium inoculation, 4) Postinoculation.

2.2 Mechanical Properties of S.G.I

Pearlitic S.G.irons are stronger but less ductile than ferritic S.G.irons. A martensitic matrix may be obtained by quenching in oil or water from 870 to 925 ^{o}C. The quenched structures are usually tempered, after hardening, to the strength and hardness levels.

Austenitic S.G.irons are highly alloyed types which remain their austenitic structure down to at least - 103 ^{o}C. These irons are of interest because of their relatively high corrosion resistance and good creep properties at elevated temperatures.[3]

Table 1. Mechanical properties of basic types of S.G.I. (Metals handbook)



TYPE	ALLOY CONTENT	TENSILE STRENGTH, PSI	YIELD STRENGTH, PSI	ELONGATION, % IN 2 IN.	BHN
Ferritic	Low	55,000	35,000	25	130
	High	90,000	70,000	12	210
Pearlitic	Low	80,000	60,000	10	200
	Low†	130,000	90,000	7	275
	High	130,000	110,000	2	275
Quenched	...	100,000	80,000	10	215
		150,000	130,000	2	320
Austenitic	‡	60,000	30,000	40	130
	§	60,000	40,000	10	160

2.3 Melting Process of S.G.Iron

2.3.1 Charge Materials

High quality productions require carefully selection of charge materials and melting process. Pig iron, steel scrap, spheroidal graphite iron return scrap are used as charge materials.

Charge materials should be provided that properties:
- chemical composition ranges
- free inhibiting trace elements
- favourable price
- continuous supply

2.3.2 Furnaces

All types of metal melting furnaces can be used for foundry operations; foundry requirements are sometimes unique and one or more another type of furnace may be best for a particular operation.

The choice of furnace may be dictated by,

1) considerations of initial cost,
2) relative average cost of maintenance and repair,
3) cost of melting,
4) melting speed and efficiency,
5) composition and melting temperature.

Induction Furnaces

Most of the modern foundry use induction furnace for melting process of S.G.iron. Induction furnaces are preferred because of high melting speed, easy composition and temperature control.

Many types and size of induction melting units are available; the most versatile type is the coreless induction furnaces. The principles of operation are essentially the same in all units.

The charge is placed inside the crucible and current is applied; more metal is charged as the melting is proceeds. Metallic charge or part of it is surrounded by an electrical conductor coil through which alternate current passes. This current creates a magnetic field which in turn results in eddy currents in the metal core to be heated. Due to resistance part of the electrical energy represented by the eddy currents is transformed into heat energy, raising the temperature of the metallic charge. The rate of energy input is dependent on the magnetic field density and also on the frequency of change of polarity.

The cored furnace is used primarily for non-ferrous metals in applications where relatively long periods of continuous operation are desirable. The major disadvantage of the cored induction furnace is that it requires a liquid metal "starting" charge; it therefore is not suitable for intermittent operation.

Cupola Furnaces

Most of the melting operations for cast irons are made by cupolas which are considered the cheapest method for converting cold scrap metal or pig iron to usable molten iron.Cupola which is a typical shaft furnace, is similar to blast furnace. Fuel for cupola is preferably high quality and low sulphur coke, antracite coal or carbon briquettes.

Cupola furnaces encompass the following basic types, refractory lined (or unlined), acid, neutral, basic slag, cold or hot blast back or front slagging with or without oxygen enrichment.

Type of slag is very effective on quality of iron. C and S contents are important for production of S.G.iron. Therefore, type of slag should be carefully selected. Slag basicity is defined as [4]

$$\frac{CaO(\%) + MgO(\%)}{SiO2(\%)} \qquad (1)$$

Table 2. Comparison of acid cupola with basic cupola .

	Acid Cupola	Basic Cupola
Carbon	Preferable with controls, a sufficiently high C, content is maintained.	As a rule C content is too high excepting thin castings, resulting in graphite flotation.
Sulphur	Too high without desulphurization prior to spheroidization treatment	low, definitely preferable
Si loss	Preferable, usually about 100 %	high, usually 20-35 %

Table 2. (cont'd)

Charge	Pig iron is required	Pig iron is not necessarily required, preferable
Magnesium	Usually more Mg is required	Preferable, less Mg required
Refractory Cost	Low usually preferably	Relatively high, but lower consumption.
Coke Consumption	Low	Higher
Melt Rate	High	lower
Iron Cleanliness	somewhat inferior	somewhat preferable
Tapping Temperature	Preferably, usually higher	Low, superheating unit is usually necessary

2.4 Desulphurization

Excess free sulphur in gray iron increases chill depth rapidly, decreases fluidity, and decreases the effectiveness of inoculation. The spheroidizer elements can combine with S, forming sulphides or complexes. Therefore, S content, which below the 0.01% is advisable. If content of S is more than 0.01%, desulphurization process will be necessary.

2.4.1 Methods of Desulphurization

Molten iron can also be desulphurized by bringing in into contact with soda ash, calcium carbide, or quicklime(calcium oxide). The efficiency and speed of desulphurization increase with area of interface contact. The amount of sulphur removed depends on the type and amount of desulphurizer used, the contact time, and the method creating interface.

The chemical reactions between S and desulphurizing agents can be described as

$$CaO + S + C = CaS + CO \qquad (2)$$

$$CaC_2 + S = CaS + 2C \qquad (3)$$

$$Na_2CO_3 + S + 2C = Na_2S + 2CO \qquad (4)$$

Desulphurization efficiency increases with increasing temperature and slag basicity.[4]

2.5 Inoculation

An inoculant is an addition made to a melt, usually late in the melting operation, which alters the solidification structure of the cast metal. The process is distinct from alloy additions made to the molten iron for the sole purpose of affecting the chemical composition of the melt.

Some common inoculation treatments include grain refinement of aluminium and magnesium alloys, graphitization of gray iron, and the use of magnesium or cerium to produce spheroidal graphite iron.

Commercial inoculants contain various amounts of carbon, silicon, chromium, magnesium, calcium titanium, zirconium, aluminium, barium and strontium.

The effect of an inoculant depends on the type and amount used, the temperature and condition of the molten iron at the time of addition, and the amount of time that elapses between inoculation ant the pouring of the casting.[4]

2.5.1 Roles of Inoculation on Spheroidization

Addition of some spheroidizer elements cause the graphite flakes to form in shape of spheres rather than flakes. This microstructural changing can be seen on Figure 3a and 3b.

When the carbon equivalent level is below the eutectic ratio of 4.3 the first crystals to form from the molten cast iron are these of austenite, containing 2% of carbon. Thus the concentration of carbon in the remaining liquid is raised progressively until it reaches the 4.3 eutectic level. At this stage the eutectic begins to grow at numerous points called nuclei.

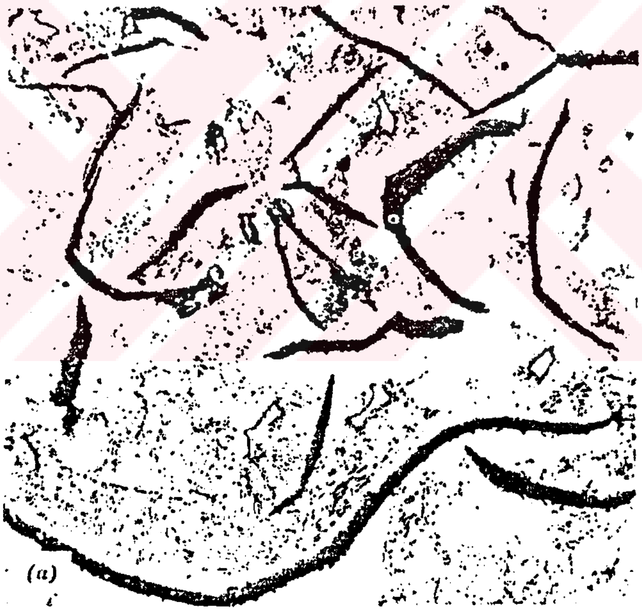


Figure 3.a Gray cast iron. Graphites as flakes [5].

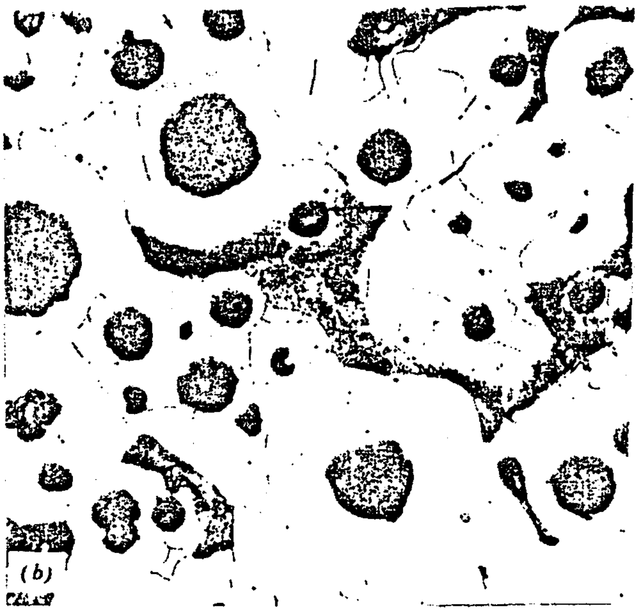


Figure 3.b Spheroidal graphite iron. Graphite as spheres [5].

From these nuclei the graphite flakes grow in clusters known as eutectic cells, until they touch one another and all the liquid has solidified. Figure 4 represents a cluster of graphite flakes in a single eutectic cell.[4]

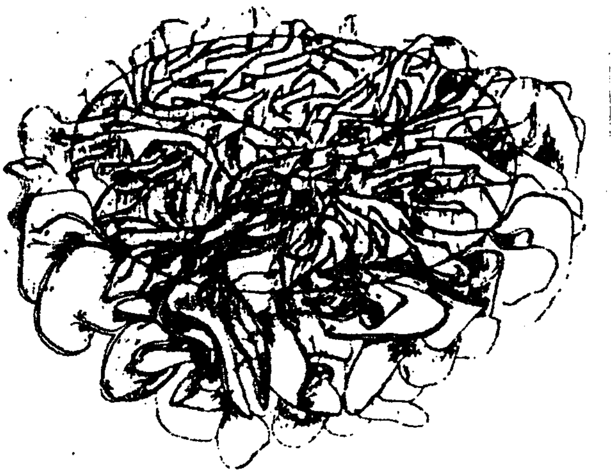


Figure 4. A cluster of graphite flakes in a single eutectic cell [4].

The growth process of spheroidal graphite iron can be thought of as being quite similar to gray cast iron except that the interdendritic liquid solidifies as spheres of graphite rather than eutectic colonies. These spheres are frequently called nodules or spherulites. The amount of supercooling for spherulite growth is larger than for eutectic cell and the number of spherulites per cubic centimeter is roughly 200 times greater than the eutectic cells of gray iron. The spherulite itself is essentially pure solid graphite, which is a spheroidal aggregate composed of a radially growing array of graphite crystallites originating at a common center. Figure 5(a) presents a chematic illustration of the appearance of the spherulites. The individual

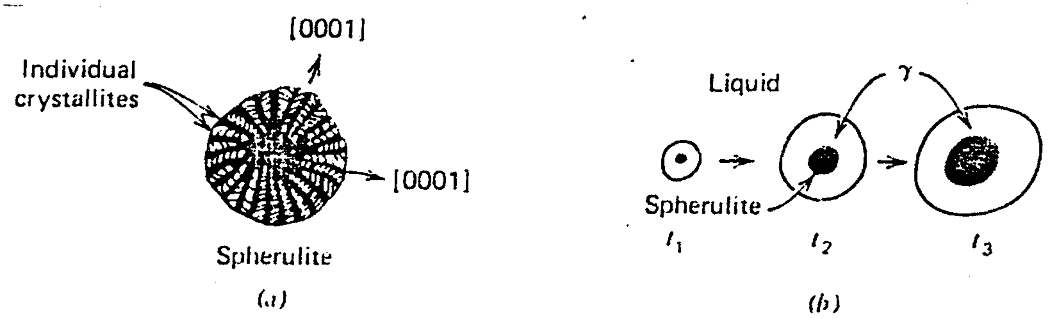


Figure 5 a) A graphite spherulite (nodule) in spheroidal cast iron. b) Growth of a spherulite (nodule) illustrating the surrounding austenite [5].

crystallites grow outward in such a manner as to maintain their C axis parallel to the growth direction (graphite has a hexagonal crystal structure). Hence the surface of the spherulite is composed of the basal planes of the graphite crystallites, and beautiful SEM pictures of these surfaces have recently become available.(5)

At the present there are three areas in which we do not understand the solidification of spheroidal graphite iron:

1. Very little is known about how the spherulites nucleate.

2.The flakes in gray iron are known to grow with their a axis parallel to the growth direction, whereas the graphite is growing parallel to the C axis in spherulite growth. The elements Mg and Ce are able to force this change in growth axis, but we do not understand how. Graphite grows with a faceted interface, and it is apparent that if solute atoms tended to absorb preferentially at the edges they could poison this type of growth. It should be clear that our understanding of the graphite morphology depends on understanding the kinetics of the interface reactions that occur when graphite cystallizes from the melt.

3. When one examines as-cast nodules it is usually observed that they are surrounded by a layer of iron. Very good experimental evidence indicates that the spherulite is surrounded by a shell of austenite and that both the spherulite and shell grow simultaneously from the liquid as shown in Figure 5(b). However this made of growth is hard to explain theoretically because, all of the carbon added to the spherulite must diffuse through the austenite shell. This is a slow process and, also, one would except tremendous pressures in the shell due to the volume increase as the graphite forms. New theories have been proposed to account for these difficulties, but it is clear that much still to be learned about one of our most common metals, cast iron.(5)

2.5.2 Importance of postinoculation

Postinoculation, which is made after Mg inoculation, is a kind of inoculation treatment. Most founderymen producing spheroidal graphite irons are quite convinced of the importance of proper postinoculation; however, great differences exist from foundry to foundry in the amount and type of inoculant used, base-silicon percentage and method and time of addition.

The effects of postinoculation are twofold. There is a composition effect primarily related to the Si addition and a graphite nucleation effect.

Effects of postinoculation on microstructure can be seen on Figure 6 and Figure 7.

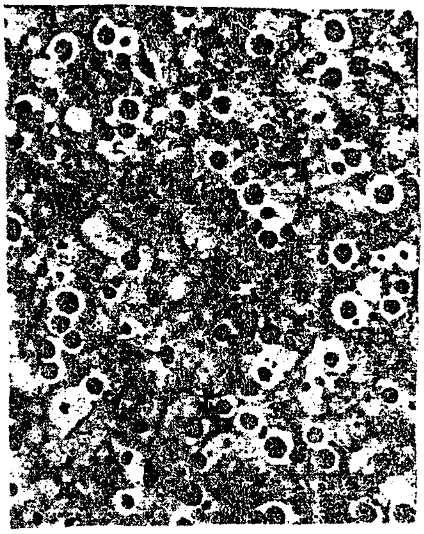


Figure 6.Without postinoculation microstructure shows pronounced as-cast acicular carbides.[14]

Figure 7.Postinoculation of metal stream with with ferrosilicon produces carbide-free microstructure.[14]

2.5.3 Parameters of Inoculation and Postinoculation

2.5.3.1 Effects of the Temperature

Inoculating efficiency deteriorates with increasing iron temperature. While inoculating temperature increases from 1315 to 1537 °C, the nodules per mm^2 decrease from 120 to 20. (6)

2.5.3.2 Effects of Time

Time is a very important parameter of inoculation.

Time between melting of metal and inoculation, and inoculation and pouring have strong influence on casting microstructure.

2.5.3.3 Effects of Chemical Composition

Silicon: Silicon is a basic component of cast iron. In the as-cast condition, increasing Si content increases ferrite/pearlite ratio, impact transition temperature, and decreases measured impact energy.

Effects of Si are shown on Figure 8, Figure 9, Figure 10, Figure 11.

Final silicon levels range from 2.0% to 3.0%. Magnesium has a greater degree of solubility in irons of higher silicon content. Silicon contents of up to 6% are used for increasing heat resistance up to 760 ^{o}C.[4]

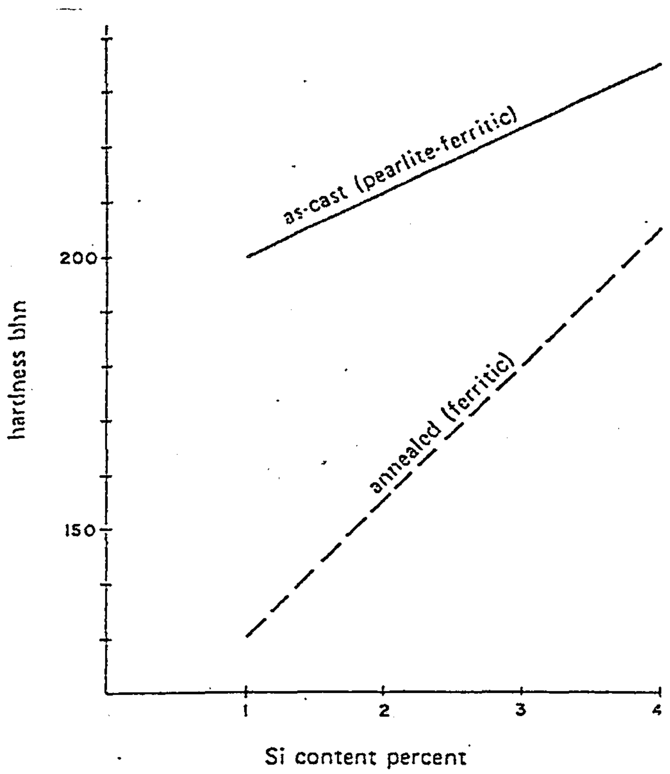


Figure 8. Hardening effect of Si on as-cast and annealed unalloyed spheroidal graphite iron.[6]

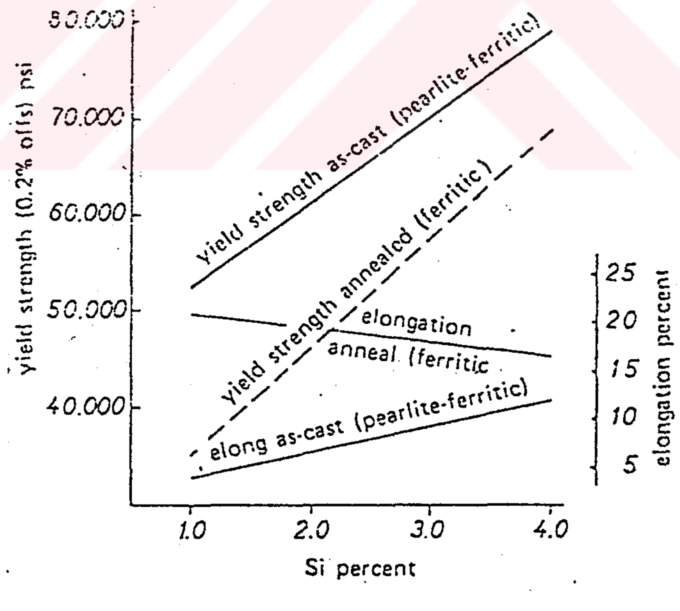


Figure 9. Effect of Si on Yield strength and elongation on as-cast and annealed spheroidal graphite iron [6].

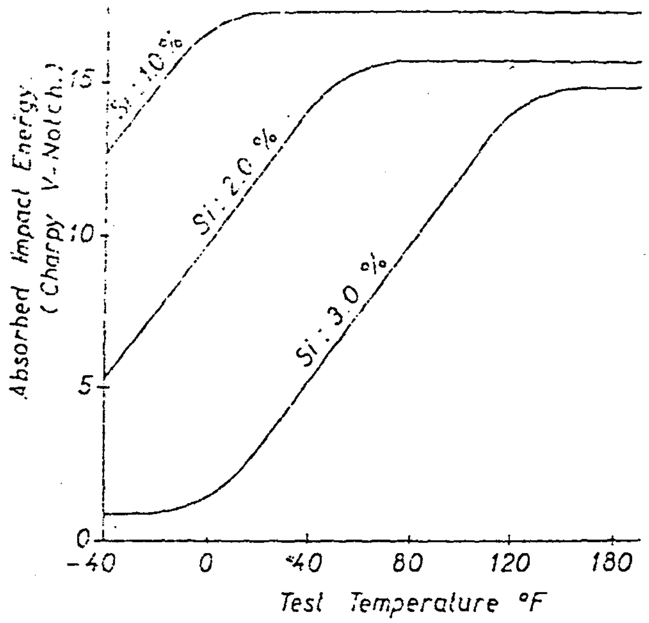


Figure 10. Impact energy at various temperatures for three

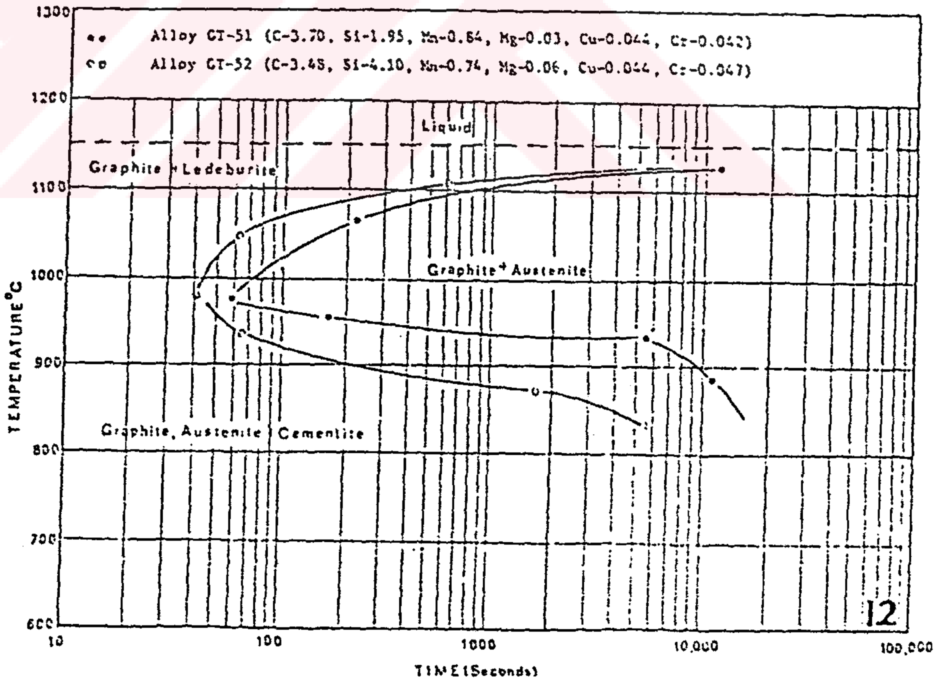


Figure 11. Influence of Si on the TTT curve for decomposition of cementite in S.G.I. [10]

Sulphur: Sulphur is held as low as possible since magnesium and rare-earth metals react first with sulphur. Content of sulphur in S.G.iron should be maximum 0.02%.

Phosphorous: The effect of P on the flowability of molten cast iron is very important. In hypereutectic irons an increase of 0.2% P improves flowability equal to addition of 0.1% C or 15 C rise in temperature.[6]

Magnesium: Minimum magnesium content of a spheroidal graphite iron should be between the values of 0.02% and 0.06%. An optimum value of magnesium to good spheroidization is 0.04%. During magnesium addition, magnesium fading must be calculated. Because of the low boiling point, magnesium fading may be very much.[4]

Manganese:Manganese can have a range from 0.15 to 0.80% but for maximum ductility and fast ferritization by heat treatment the manganese should be low. Manganese on the high side of the range is desirable for highest as cast strength and best response to hardening heat treatments.

Nickel: Nickel increases the strength properties of the as cast material but tends to retard ferritization as it is a pearlite stabilizer.

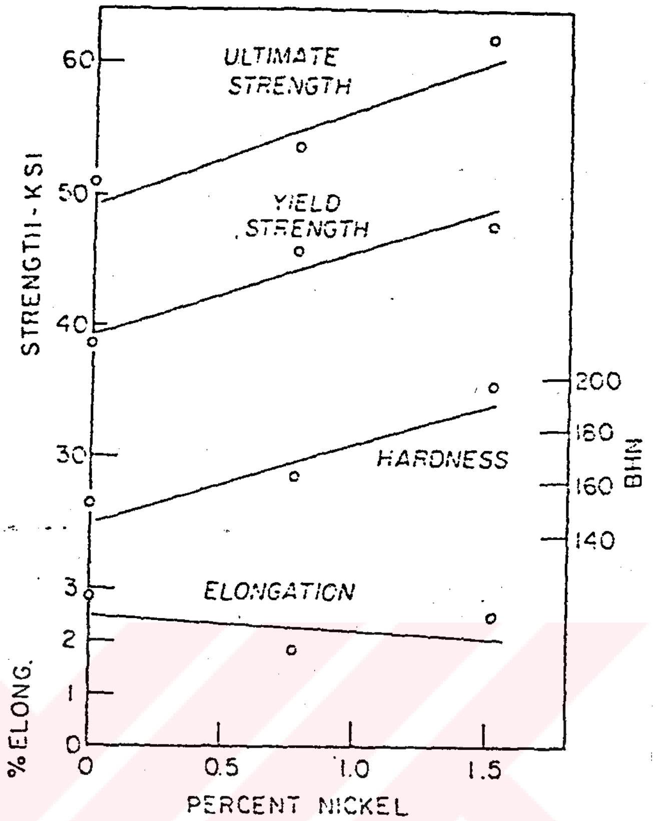


Figure 12. Effect of nickel on as-cast mechanical properties of cast iron containing vermicular graphite.[11]

Chromium: Chromium is not a usual addition to S.G.iron as at about 0.15% it makes castings difficult to ferritize.

Molybdenum: Molybdenum increases the strength and hardness of heavy sections and improves strength at elevated temperature. It is added up to 1.0%.

Copper: Copper may be used up to 1.0% as it stabilizes pearlite and gives added strength and hardness but inhibits ferritization by annealing. It has the adverse effect of increasing the effect of trace elements which encourage the formation of degenerate graphite shapes.

2.6 Structure of Cast Iron With Vermicular Graphite

Vermicular graphite can be found in cast iron with spheroidal graphite. This is an in between structure between spheroids and flakes. In contrast to spheroidal graphite found in the eutectic crystallization stage surrounded by a closed austenite shell [8,9], eutectic crystallization of vermicular graphite colonise proceeds with one-sided contact of the graphite with the melt. This causes a marked difference in the distribution of chemical elements and in the features of vermicular graphite and spheroidal graphite [7].

The compact form of vermicular graphite and their independent structures provide high mechanical properties for cast iron in the casting condition, close to the properties of cast iron with spheroidal graphite.[7]

2.7 Spheroidization

Spheroidization is a process in which an inoculant (spheroidizer) is added to molten gray iron for the purpose of affecting the shape, size and distribution of graphite in the casting.

An inoculant (spheroidizer) (Mg,Ce) can be introduced into the molten iron in several ways, and in several locations in the process sequence. It can be added to the metal in the forehearth, in the transfer ladle, in the pouring ladle, and in the mold.

2.7.1 Spheroidization Treatments

Forehearth Inoculation

The inoculant is usually added in the forehearth, or holding ladle, by an automatic feeding device. When the inoculant is fed into the steam of metal flowing into the forehearth, it is thoroughly distributed and loss of effectiveness through flotation or sticking to the sides of the ladle is minimised.

The feeding device may consists merely of a small funnel fastened directly over the end of the cupola spout to deliver the inoculant into the stream of molten iron, or it may be a feed belt that runs at a speed corresponding to the rate of iron flow from the spout. When iron is inoculated in the forehearth, the amount of inoculant must be sufficient to compensate for some fading of effectiveness, because it may be some time before the iron is poured into molds.

Transfer-Ladle Inoculation

Inoculation is more often added in the transfer ladle than in the forehearth. It is added to the stream as the metal is transferred, usually by means of a long-handle cup. For a given application, less inoculant is needed when added to the transfer ladle when added to the forehearth, because time to pouring of the casting is less and consequently there is less fading of the inoculant. There should be a "heel" of iron, several inches deep in the ladle before the inoculant is added. The heel prevents the inoculant from being carried directly to the bottom or side of the ladle and fusing to the walls, or from being trapped by the slaggy amorphous coating that is present in all ladles.

Pouring-Ladle Inoculation

Frequently, an inoculation made in the forehearth or in the transfer ladle is revitalised by adding more inoculant in the pouring ladle. The inoculant is added to the stream of metal being poured from the transfer ladle into the pouring ladle. Usually, the amount of inoculant needed for pouring-ladle additions is small compared to the amount used for forehearth or transfer-ladle additions. After inoculation in the pouring-ladle,the slag must be skimmed before the iron is poured; skimming is required because, if mixing has been incomplete, the inoculant may float on the surface of the molten metal, and some inoculant may be carried into the mold, causing defective castings.

In-Mold Inoculation

In-mold inoculation is less common for gray iron than for spheroidal graphite iron, but it is sometimes used. The technique consists of adding small amounts of extrafine inoculating material at the sprue or the runner after the mold is closed and before the casting is poured.For in-mold inoculation, the amount, grain size, and melting point of the inoculant are important, because there is little time available for the inoculant to dissolve. It is recommended that the castings be poured with iron that is as high as practical in temperature. Undissolved particles of inoculant are less likely to remain if the iron is near the maximum pouring temperature.

2.7.2 Fading of Inoculants

The effects of inoculation, which is at a maximum immediately after the addition has been made, is not permanent. The loss of the inoculating effect, which occurs without any noticeable change in metal composition, is known as "fading". The rate of fading follows a typical exponential curve, and the maximum rate of fade occurs immediately after the addition is made. In practice, about half of the inoculating effect produced is likely to be lost within five minutes of adding the inoculant. Not all inoculants fade to an equal extent, and variations occur in the initial potency and inoculation practice.

The view that very little fading occurs in the first five or ten minutes after inoculation, and that some inoculants do not develop their full effect until several minutes have elapsed after their addition, has little experimental support [2]. The amount of published information which compares, under similar conditions, the fading characteristics of different inoculants,is limited; eventhough, periodically, claims are made that a particular inoculant is more resistant to fade than others [2].

2.7.2.1 FADING OF MAGNESIUM TREATMENT

The loss of Mg and volatilisation during the magnesium treatment process is considerable; the reaction efficiency, measured in terms of Mg recovery, is therefore quite low. Depending on the particular technique used, magnesium recovery in the spheroidizing treatment ranges between 20 and 60 percent.

Some researches were aimed at increasing the efficiency of magnesium additions to approach 100 percent recovery of Mg. One method of improving reaction efficiency is to eliminate the boiling of magnesium during addition by using as the treatment alloy a Mg-Fe-Si alloy whose Mg partial pressure at cast iron working temperature (1370-1510 ^{o}C) is less than or equal to one atmosphere.

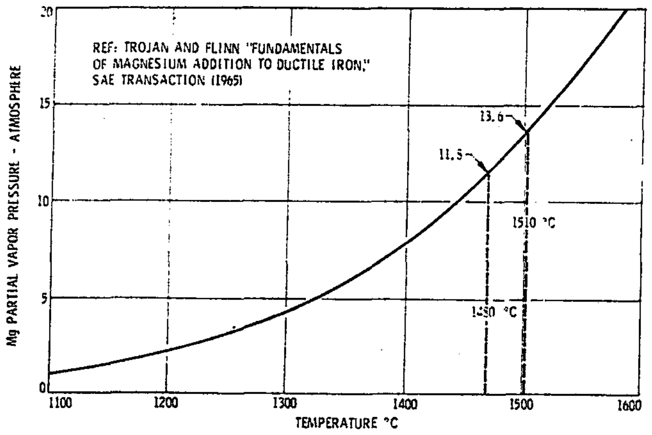


Figure 13. Vapor pressure of pure Mg at cast iron melting temperature [12].

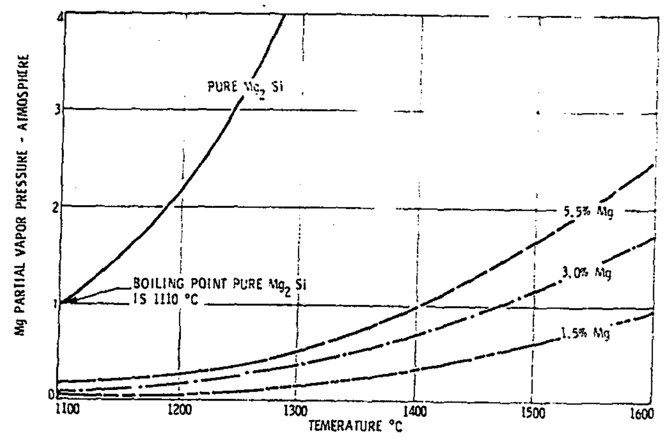


Figure 14. Calculated vapor pressure of Mg-Fe-Si alloys at cast iron melting temperature.[12]

Magnesium treatment fading can be investigated at three points:

1. fading of magnesium
2. fading of nodule count
3. fading of nodularity

A schematic representation of fading of Mg content and nodularity is presented in Figure 15.

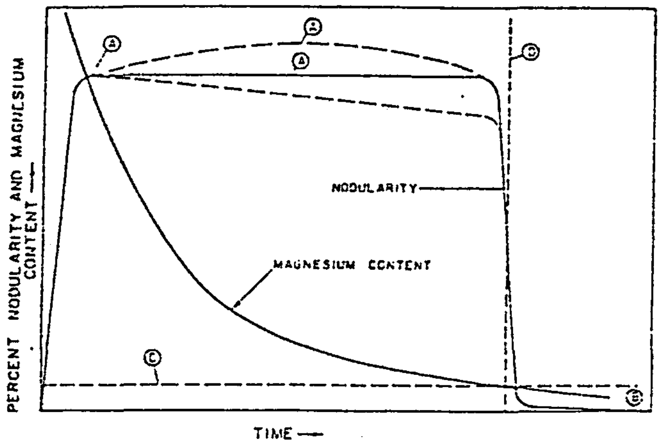


Figure 15. Relative loss of magnesium content and nodularity with time [13].

Appreciable nodularity fade however does not occur until a critical level of nodularity (A) is achieved shortly after treatment. This level may persist during the given holding time, or it may decrease or increase slightly depending upon the specific details of the treatment process. When the critical level of Mg is reached, nodularity drops rapidly to near zero(B). Where the initial Mg:S is greater than 3.0 nodularity fade will fall from 100% nodularity to near zero, but where 3.0>Mg:S>1.0 considerable fade might be realised prior to reaching the equivalent time D.[13]

As the Mg:S levels in the iron change during holding, other aspects of the fadenig process become apparent. A shematic summary is present in Figure 16. This graph of Mg vs S, as analysed in the iron at any given time following treatment, is divided into three areas:

1. A region of Mg:S less than 1.0 where the treatment process of this study resulted in the formation of graphite which was predominantly flake-shaped.
2. A region of Mg:S greater than 3.0, where the predominant graphite form was spheroidal.
3. An intermediate region, where spheroidal graphite and vermicular graphite were observed.

Magnesium fading increase with :

1. increasing holding temperature
2. increasing initial magnesium content.
3. the treated iron is being stirred.

Magnesium fading decreases with:

1. increasing lining and slag basicity.
2. increasing quantity of treated iron.

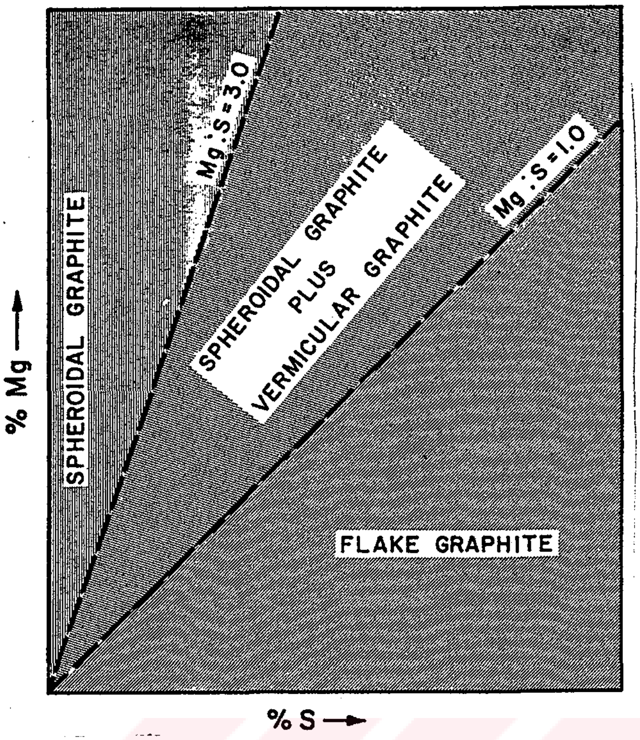


Figure 16. Schematic diagrams summarizing the magnesium treatment fading. [13]

CHAPTER III

EXPERIMENTAL PROCEDURE AND RESULTS

In this chapter, the experiments performed to determine the influence of the some major parameters of postinoculation will be explained.

Apparatus

The following apparatus has been used in the experiments.

1) Melting furnace.
2) Sand molding machines.
3) Optic pyrometer.
4) Thermocouple.
5) Belt surfacer.
6) Mechanical polishing device.

Procedure

1) Charge preparation.
2) Melting operation.
3) Casting.
4) Cast analysis.

It was planned to investigate the effect of postinoculation time and the time passing between Mg inoculation and postinoculation on the graphite structure.

Experiment 1:

Chemical composition of sorel pig: % C= 4.3
% Si= 0.09
% Fe= 94.5

Chemical composition of steel: % C= 0.2
% Si= 0.3
% Fe= 99.2

Chemical composition of Fe-Si: % Si= 65
% Fe= 35

Chemical composition of Fe-Si-Mg: % Mg= 5
% Si= 45
% Fe= 50

Desired chemical composition of product : %C= 3.8
%Si= 2.1
%Mg= 0.05

Calculated amounts of charge materials :

Sorel pig= 1700 gr
Steel= 161.5 gr
Fe-Si= 40.8 gr
Fe-Si-Mg= 28.9 gr
CaO= 17gr (for desulphurization)

The steps of experiment 1.:

1. Charge materials was weighed.
2. Sorel pig and steel were placed in induction furnace.
3. Charge was melted.
4. Temperature was measured as 1350 °C.
5. CaO desulphurization was done by plunging.
6. Temperature was measured as 1400 °C.
7. Fe-Si-Mg was plunged into liquid metal for Mg inoculation.
8. After Mg inoculation, liquid metal was waited at 1350° C for one minute.
9. Postinoculation was made by using FeSi.
10. Pouring temperature was measured as 1300 °C.
11. Casting of liquid metal into 5 sand molds was performed with 2 minutes time intervals.

Experiment 2.1:

Chemical composition of sorel pig: % C= 4.3
% Si= 0.09
% Fe= 94.5

Chemical composition of steel: % C= 0.2
% Si= 0.3
% Fe= 99.2

Chemical composition of Fe-Si: % Si= 65
% Fe= 35

Chemical composition of Fe-Si-Mg: % Mg= 5

% Si= 45

% Fe= 50

Desired chemical composition of product : %C= 3.8

%Si= 2.1

%Mg= 0.05

Calculated amounts of charge materials :

Sorel pig= 850 gr

Steel= 80.75 gr

Fe-Si= 20.4 gr

Fe-Si-Mg= 14.45 gr

CaO= 8.5 gr (for desulphurization)

The steps of experiment 2.1.:

1. Charge materials was weighed.
2. Sorel pig and steel were placed in induction furnace.
3. Charge was melted.
4. Temperature was measured as 1350 °C.
5. CaO desulphurization was done by plunging.
6. Temperature was measured as 1400 °C.
7. Fe-Si-Mg was plunged into liquid metal for Mg inoculation.
8. After Mg inoculation, liquid metal was waited at 1350 °C for two minutes.
9. Postinoculation was made by using FeSi.
10. Pouring temperature was measured as 1300 °C.
11. Casting of liquid metal into sand molds was performed 2 minutes after postinoculation.

Experiment 2.2:

Chemical composition of sorel pig: % C= 4.3
% Si= 0.09
% Fe= 94.5

Chemical composition of steel: % C= 0.2
% Si= 0.3
% Fe= 99.2

Chemical composition of Fe-Si: % Si= 65
% Fe= 35

Chemical composition of Fe-Si-Mg: % Mg= 5
% Si= 45
% Fe= 50

Desired chemical composition of product : %C= 3.8
%Si= 2.1
%Mg= 0.05

Calculated amounts of charge materials :

Sorel pig= 1050 gr
Steel= 99.75 gr
Fe-Si= 25.2 gr
Fe-Si-Mg= 17.85 gr
CaO= 10.5 gr (for desulphurization)

The steps of experiment 2.2:

1. Charge materials was weighed.
2. Sorel pig and steel were placed in induction furnace.
3. Charge was melted.

4. Temperature was measured as 1350 °C.
5. CaO desulphurization was done by plunging.
6. Temperature was measured as 1400 °C.
7. Fe-Si-Mg was plunged into liquid metal for Mg inoculation.
8. After Mg inoculation, liquid metal was waited at 1350 °C for four minutes.
9. Postinoculation was made by using FeSi.
10. Pouring temperature was measured as 1300 °C.
11. Casting of liquid metal into sand molds was performed 2 minutes after postinoculation.

Experiment 2.3:

Chemical composition of sorel pig: % C= 4.3
% Si= 0.09
% Fe= 94.5

Chemical composition of steel: % C= 0.2
% Si= 0.3
% Fe= 99.2

Chemical composition of Fe-Si: % Si= 65
% Fe= 35

Chemical composition of Fe-Si-Mg: % Mg= 5
% Si= 45
% Fe= 50

Desired chemical composition of product : %C= 3.8
%Si= 2.1
%Mg= 0.05

Calculated amounts of charge materials :

Sorel pig= 950 gr

Steel= 90.25 gr

Fe-Si= 22.8 gr

Fe-Si-Mg= 16.15 gr

CaO= 9.5 gr (for desulphurization)

The steps of experiment 2.3:

1. Charge materials was weighed.
2. Sorel pig and steel were placed in induction furnace.
3. Charge was melted.
4. Temperature was measured as 1350 °C.
5. CaO desulphurization was done by plunging.
6. Temperature was measured as 1400 °C.
7. Fe-Si-Mg was plunged into liquid metal for Mg inoculation.
8. After Mg inoculation, liquid metal was waited at 1350 °C for six minutes.
9. Postinoculation was made by using FeSi.
10. Pouring temperature was measured as 1300 °C.
11. Casting of liquid metal into sand molds was performed 2 minutes after postinoculation.

Experiment 2.4:

Chemical composition of sorel pig: % C= 4.3

% Si= 0.09

% Fe= 94.5

Chemical composition of steel: % C= 0.2
% Si= 0.3
% Fe= 99.2

Chemical composition of Fe-Si: % Si= 65
% Fe= 35

Chemical composition of Fe-Si-Mg: % Mg= 5
% Si= 45
% Fe= 50

Desired chemical composition of product : %C= 3.8
%Si= 2.1
%Mg= 0.05

Calculated amounts of charge materials :

Sorel pig= 850 gr
Steel= 80.75 gr
Fe-Si= 20.4 gr
Fe-Si-Mg= 14.45 gr
CaO= 8.5 gr (for desulphurization)

The steps of experiment 2.4. :

1. Charge materials was weighed.
2. Sorel pig and steel were placed in induction furnace.
3. Charge was melted.
4. Temperature was measured as 1350 °C.
5. CaO desulphurization was done by plunging.
6. Temperature was measured as 1400 °C.

7. Fe-Si-Mg was plunged into liquid metal for Mg inoculation.
8. After Mg inoculation, liquid metal was waited at 1350 °C for eight minutes.
9. Postinoculation was made by using FeSi.
10. Pouring temperature was measured as 1300 °C.
11. Casting of liquid metal into sand molds was performed 2 minutes after postinoculation.

Experiment 2.5:

Chemical composition of sorel pig: % C= 4.3
% Si= 0.09
% Fe= 94.5
Chemical composition of steel: % C= 0.2
% Si= 0.3
% Fe= 99.2
Chemical composition of Fe-Si: % Si= 65
% Fe= 35
Chemical composition of Fe-Si-Mg: % Mg= 5
% Si= 45
% Fe= 50
Desired chemical composition of product : %C= 3.8
%Si= 2.1
%Mg= 0.05

Calculated amounts of charge materials :

Sorel pig= 1100 gr

Steel= 104.5 gr
Fe-Si= 26.4 gr
Fe-Si-Mg= 18.7 gr
CaO= 11 gr (for desulphurization)

The steps of experiment 2.5.:

1. Charge materials was weighed.
2. Sorel pig and steel were placed in induction furnace.
3. Charge was melted.
4. Temperature was measured as 1350 °C.
5. CaO desulphurization was done by plunging.
6. Temperature was measured as 1400 °C.
7. Fe-Si-Mg was plunged into liquid metal for Mg inoculation.
8. After Mg inoculation,liquid metal was waited at 1350 °C for ten minutes.
9. Postinoculation was made by using FeSi.
10. Pouring temperature was measured as 1300 °C.
11. Casting of liquid metal into sand molds was performed 2 minutes after postinoculation.

Metallographic Studies

The soft nature of the graphite phase, in combination with the relatively hard matrix, gives difficulties during preparation of spheroidal cast iron for metallographitic examination.

Surface damage that occurs during mechanical operations after the casting was eliminated by grinding and polishing.

Grinding was accomplished by abrading the specimen surface through a sequence of operations. Emery paper number which were used, can be sequenced 120, 220, 320, 400, 500, and 600. All grinding operations were made wet.

Polishing is the final step of the production of the flat surface. Mechanical polishing was done by using of cloth-covered laps and suitable polishing abrasives. Aluminium oxide was used as abrasives during polishing operations.

Metallographic etching were used to clear particular structural characteristics of an as-polished metal. All specimens were etched by 2 % nital and then were cleaned by water and alcohol and finally were dried by warm air.

Experimental Results

Experiment 1:

Table 3. Chemical composition of final product (%) of exp. 1.

C	Mn	Si	P	S	Cr	Ni
4.18	0.075	2.050	0.01	0.006	0.023	0

Ti	Fe	Al	Sn	Cu
0	93.56	0.146	0.001	0.030

Experiment 2.1

Table 4. Chemical composition of final product (%) of exp. 2.1.

C	Mn	Si	P	S	Cr	Ni
3.409	0.112	2.012	0.008	0.011	0.036	0.035

Ti	Fe	Al	Sn	Cu
0	94.14	0.075	0.001	0.050

Experiment 2.2

Table 5. Chemical composition of final product (%) of exp. 2.2.

C	Mn	Si	P	S	Cr	Ni
4.39	0.085	2.015	0.01	0.007	0.043	0.002

Ti	Fe	Al	Sn	Cu
0	94.11	0.045	0.002	0.065

Experiment 2.3

Table 6. Chemical composition of final product (%) of exp. 2.3.

C	Mn	Si	P	S	Cr	Ni
4.35	0.08	2.6	0.021	0.006	0.043	0.003

Ti	Fe	Al	Sn	Cu
0.003	93.56	0.053	0.001	0.050

Experiment 2.4

Table 7. Chemical composition of final product (%) of exp. 2.4.

C	Mn	Si	P	S	Cr	Ni
3.88	0.08	2.67	0.019	0.010	0.019	0.001

Ti	Fe	Al	Sn	Cu
0.001	94.129	0.08	0.075	0.3

Experiment 2.5.

Table 8. Chemical composition of final product (%) of exp. 2.5.

C	Mn	Si	P	S	Cr	Ni
3.82	0.054	2.15	0.02	0.0036	0.033	0.002

Ti	Fe	Al	Sn	Cu
0.002	94.02	0.034	0.015	0.024

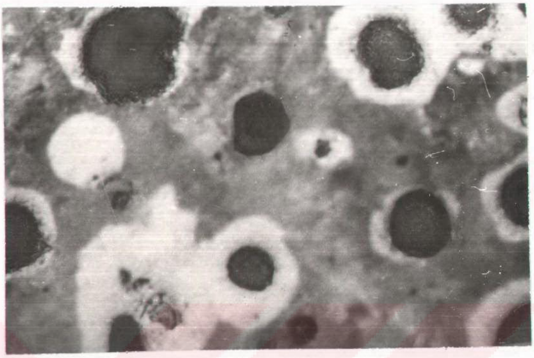


Figure 17. Sample 1.1. Etched with 2 % nital. X 200.
Specimen 1.1 was poured 2 minutes after postinoculation.

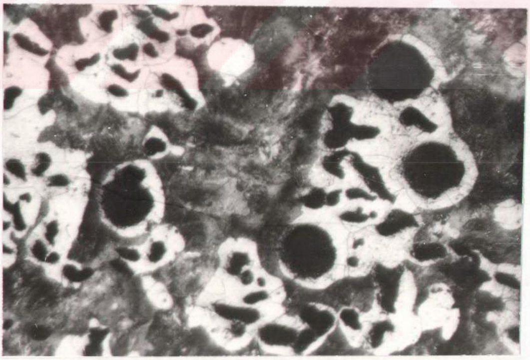


Figure 18. Sample 1.2. Etched with 2 % nital. X 200.
Specimen 1.2 was poured 4 minutes after postinoculation.

Figure 19. Sample 1.3. Etched with 2 % nital. X 200.

Specimen 1.3 was poured 6 minutes after postinoculation.

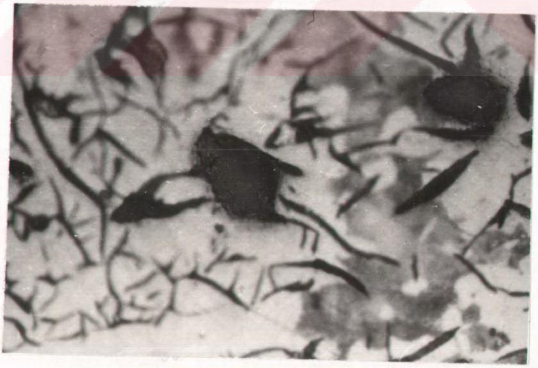


Figure 20. Sample 1.4. Etched with 2 % nital X 200.

Specimen 1.4 was poured 8 minutes after postinoculation.

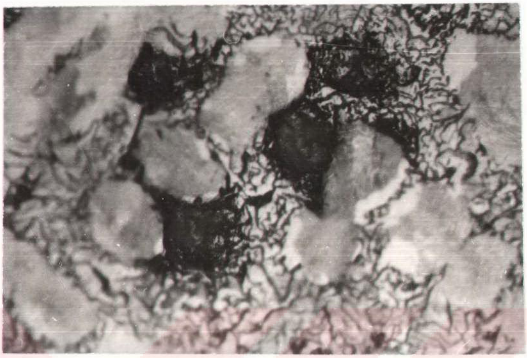


Figure 21. Sample 1.5. Etched with 2 % nital. X 200.

Specimen 1.5 was poured 10 minutes after postinoculation.

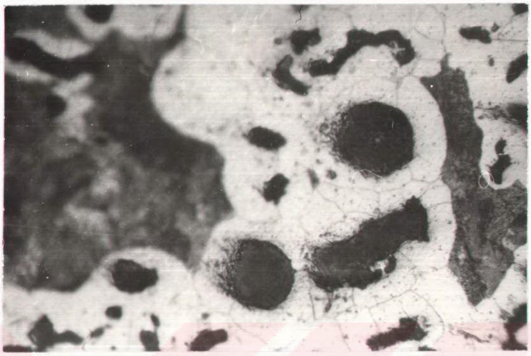


Figure 22. Sample 2.1. Etched with 2 % nital. X 200. Specimen 2.1 was postinoculated 2 minutes after Mg-inoculation.

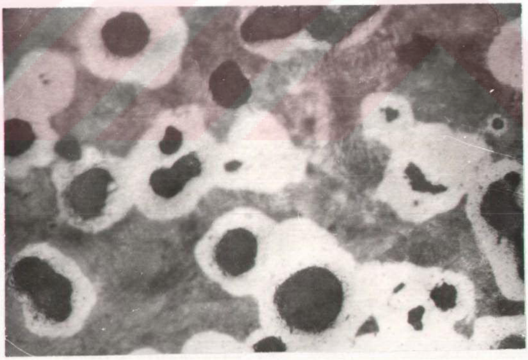


Figure 23. Sample 2.2. Etched with 2 % nital. X 200. Specimen 2.2 was postinoculated 4 minutes after Mg-inoculation.

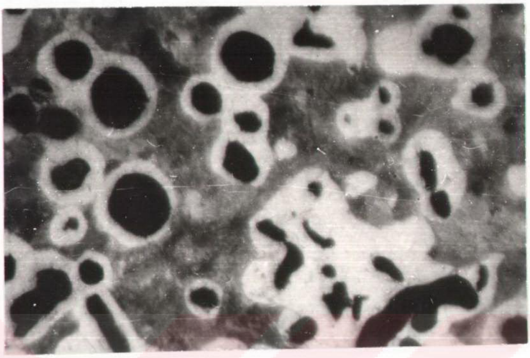


Figure 24. Sample 2.3. Etched with 2 % nital. X 200. Specimen 2.3 was postinoculated 6 minutes after Mg-inoculation.

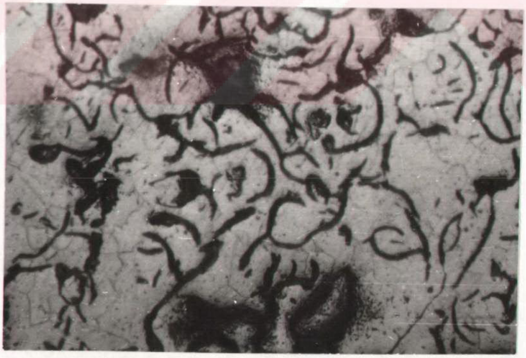


Figure 25. Sample 2.4. Etched with 2 % nital. X 200. Specimen 2.4 was postinoculated 8 minutes after Mg-inoculation.

Figure 26. Sample 2.5. Etched with 2 % nital. X 200. Specimen 2.5 was postinoculated 10 minutes after Mg-inoculation.

CHAPTER IV

DISCUSSION

In this study, 2 sets experiments which contain 6 casting operations, were performed. The first set was related with time between postinoculation and pouring, and the other set was related with time between Mg inoculation and postinoculation.

There is direct relationship between mechanical properties of S.G.I and nodule size, shape, count, distribution and matrix. The analysis of nodulization occurrence shows that nodules characteristics (size, shape, count, distribution) are the result of the effects of base iron composition, Mg inoculation, postinoculation, pouring temperature and time.

As stated in the previous chapters the basic elements which are C, Mg, Si, Al, Ni, Cr, Mo, S, Mn have a great influence on the spheroidization of graphite and the matrix of the casting. Generally Cr, Mo are carbide formers and Si, Al, Ni are graphite formers. S and Mn are two of the most important element affecting the form of the carbide particles. Therefore a small change in their contents has a powerful effect on the morphological feature of graphite. The amount of C and Si determine the ratio of austenite and graphite present in the as-cast material. These two element are basic parameters of carbon equivalent (CE). Graphite spheroidization is easier at higher CE(C%+1/3Si%).

Enhances number of graphite nuclei and more regularly shaped graphite spheroids are two important advantages of the postinoculation after Mg inoculation.

In the first experiment, it was tried to get best pouring time after postinoculation. When the postinoculation is made the influence of FeSi will immediately begin. This influence will be maximum on morphology in a few minutes after the postinoculation treatment, and also this maximum influence will disappear because of fading. Therefore the postinoculated liquid metal should be casted into the mold at the maximum influence of the inoculation.

The effectiveness of an inoculant is assessed by its initial potency and its ability to maintain this effect during the time which the metal is held while casting are being poured. When an inoculant loss its effectiveness, fading will begin. Initial potency and fading behaviour change from inoculant to inoculant. That variations in inoculating behaviour occur with changes in inoculant composition and with changes in the using conditions.

Inoculants which contain Mg, should be selected and used very carefully. Because of low boiling point of Mg, Mg loss may be very much. So effectiveness of inoculant decreases. in order to obtain efficient inoculation, Mg losses should be calculated and FeSiMg combination should be used

To point out best pouring time after postinoculation, sorel pig iron and steel were charged to the induction furnace and melted, Mg inoculated, postinoculated and then this liquid were casted into 5 sand molds in 2 minutes periods intervals.Then these specimens were studied metallographically.

In first set experiment, we can see well-shaped spheroids at the first specimen, but more and again well-shaped spheroids are present at second specimen. There is an increase in nodule count from specimen 1 to specimen 2. Nodules are surrounded by ferrite rings. After these two specimens, the nodules shapes go to bad. At the third specimen, there are flakes but these are related with spheroidal graphites. This situation are known as fading. In dependent flakes and again some spheroidal graphites can be seen at the fourth specimen. Finally the last specimen contains ferrite regions, pearlite and some degenerate spheroidal graphite.

Briefly, for the first set experiment, we can say that the most and well-shaped spheroidal graphites were obtained at the second specimen which were poured 4 minutes after the postinoculation.

In the second experiment group, it was tried to find out the best time between Mg inoculation and postinoculation. In order to find this timing, 5 casting operations were made. At all of this 5 different casting operations, sorel pig and steel were charged into induction furnace and melted, Mg inoculated and postinoculated. The times between Mg inoculation and postinoculation were 2,4,6,8 and 10 minutes. All of this liquid were casted 2 minutes after the postinoculation.

At the second set experiment, we can see spheroidal graphites and around these spheroidal graphites ferrite rings. But the main difference of these 3 specimens are on the nodule count and shape. Nodule count increase from specimen 1 to specimen 3. And also shapes of nodules become more regular. At the 4 and 5 specimens fading occur. There are flakes which are related to spheroidal graphites at specimen 4 however, these flakes become independent and bigger at specimen 5.

At the second set experiment, the most well-shaped nodules were obtained at the third specimen which are produced by a 6 minutes time interval between Mg inoculation and postinoculation.

Some deviations and errors may come from the following sources during thesis work:

1) At 1400 ^{o}C,FeSiMg was plunged in to liquid metal. During each Mg inoculation treatment, 50% of Mg loss was toleranced. But, Because of our inoculation method, Mg loss may be a bit more.

2) During all casting operations, we used same thermocouple. The calibration of thermocouple weren't made a long time. So there may be some errors in temperature values.

CHAPTER V

CONCLUSION

From the research, observations and results obtained during this study, following conclusions can be drawn:

1) Pouring time after postinoculation have a powerful effect on the microstructure of as-cast S.G.I.

2) Timing between Mg inoculation and postinoculation is very important. The efficiency of postinoculation are effected by this timing.

3) The best time for pouring after the postinoculation is 4 minutes.

4) The best time for postinoculation after the Mg inoculation is 6 minutes.

5) Mg inoculation, postinoculation and pouring should be should be within 10 minutes. But this time is greatly dependent upon the casting section size, pouring temperature and inoculation practice.

6) Postinoculation has got good influence on mechanical properties because more, well shaped and better distributed spheroidal graphite can be obtained by postinoculation.

7) Because of good influence on spheroidal graphite, all foundries should use postinoculation technique.

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