University of Northern Iowa
UNI ScholarWorks

Dissertations and Theses @ UNI
Student Work

1996

The effects of post inoculant additions on ductile iron fade

Travis James Frush
University of Northern Iowa

Let us know how access to this document benefits you

Copyright ©1996 Travis James Frush

Follow this and additional works at: https://scholarworks.uni.edu/etd

Part of the Industrial Engineering Commons, and the Metallurgy Commons

Recommended Citation

Frush, Travis James, "The effects of post inoculant additions on ductile iron fade" (1996). *Dissertations and Theses @ UNI*. 1266.
https://scholarworks.uni.edu/etd/1266

This Open Access Thesis is brought to you for free and open access by the Student Work at UNI ScholarWorks. It has been accepted for inclusion in Dissertations and Theses @ UNI by an authorized administrator of UNI ScholarWorks. For more information, please contact scholarworks@uni.edu.

Offensive Materials Statement: Materials located in UNI ScholarWorks come from a broad range of sources and time periods. Some of these materials may contain offensive stereotypes, ideas, visuals, or language.

THE EFFECTS OF POST INOCULANT ADDITIONS ON DUCTILE IRON FADE

An Abstract of a Thesis

Submitted

In Partial Fulfillment

of the Requirements for the Degree

Master of Arts

Travis James Frush

University of Northern Iowa

June 1996

ABSTRACT

This study involved the investigation of three specific, commercially available, post inoculants for ductile iron and their effect on the magnesium fading rate and chill reducing tendency in a low volume production environment. Although fading effects of ductile iron have been extensively investigated, in general, they apply to high volume production operations. Unfortunately, for the smaller foundry operations, many of the process solutions proposed for the prevention of the fading effects are not applicable or practical. In addition to the small production operations, new process technologies are also excluded from many of these process solutions. For this reason, a study was conducted to determine the influence of inoculants on the fading effects of magnesium and inoculation during extended holding of molten nodular iron.

The one-step magnesium and inoculation treatment process was utilized in this study. The master alloy was a 6% magnesium ferrosilicon, barium containing, alloy. The experimental inoculants used for the study were chosen based on their specific chemical composition. Experimental inoculant A, a foundry grade ferrosilicon 75% alloy, was chosen as the control group. Experimental inoculants B and C were chosen because of their varying levels of calcium and barium in the alloy.

Results obtained from the three experiments regarding any beneficial effect on magnesium fade, showed that there was no substantial differences between the three experimental inoculants. The residual magnesium levels, percentage nodularity and

brinell hardness values, for the three experiments, all decreased with time at comparable rates. It was demonstrated that the experimental inoculants B and C are superior to the control inoculant A in terms of their chill reducing tendency in the thin-walled test samples. The high barium and high calcium containing inoculant C, proved to be the most effective chill reducer by limiting carbide formation while maintaining acceptable nodularity in the as-cast thin-walled test castings over an extended period of time.

THE EFFECTS OF POST INOCULANT ADDITIONS ON DUCTILE IRON FADE

A Thesis

Submitted

In Partial Fulfillment

of the Requirements for the Degree

Master of Arts

Travis James Frush

University of Northern Iowa

June 1996

This Study By: Travis James Frush

Entitled: THE EFFECTS OF POST INOCULANT ADDITIONS
ON DUCTILE IRON FADE

has been approved as meeting the thesis requirement for the Degree of Masters of Arts in Industrial Technology.

6-24-96
Date
Dr. Yury S. Lerner, Chair, Thesis Committee

6/24/96
Date
Dr. Mohammed F. Fahmy, Thesis Committee Member

6/24/96
Date
Dr. John W. Swope, Thesis Committee Member

8/27/96
Date
Dr. John W. Somervill, Dean, Graduate College

TABLE OF CONTENTS

Chapter Page

LIST OF TABLES

LIST OF FIGURES

Figure Page

CHAPTER I

INTRODUCTION

Since the introduction of ductile iron, also termed as nodular or spheroidal graphite iron, in the late 1940s, extensive research studies have been conducted on the variables affecting its production. Among the problems widely documented in these studies is the fading of the magnesium treatment and inoculant effects during extended holding of the molten nodular iron. Pseudolamellar structures are the result of a combination of the fading of spheroidizing and inoculation effects (QIT-Fer et Titane Inc., 1992). Although fading effects of ductile iron has been extensively investigated since the introduction of ductile iron as a new engineering material at the 1948 annual meeting of the American Foundrymen's Society, in general, they apply to foundries equipped with high holding furnace capacities, varying from 1.5 - 20 tons (QIT-Fer et Titane Inc., 1992). Unfortunately, for the smaller foundry operations, many of the process solutions proposed for the prevention of the fading effects are not applicable or practical. In addition to the small production operations, new process technologies are also excluded from many of these process solutions. Because of the different production environments between large and small foundry operations, several process variables in the prevention of the fading effects of ductile iron need to be considered separately.

In 1995, the University of Northern Iowa was granted an opportunity to investigate a new casting process developed in a joint effort between General Motors and

Hitchner Manufacturing, Inc. The process known as the Loose Sand-Vacuum Assisted Casting (LS VAC) process incorporates pressure differential mold filling. This process involves a series of manually performed operations including the loading and unloading of molds. The molds are inserted into a specially designed cylinder and back filled with loose sand for support. The average cycle time for the loading, casting and unloading of a mold is approximately 5 to 8 minutes depending on the experience of the operator. An investigation into casting ductile iron utilizing the LS VAC process gave rise to the concern of the extended holding of molten nodular iron within the process capabilities available at the university.

The main focus of the research work was concentrated on the utilization of commercially available post-inoculants in extending the fading effects of magnesium and inoculation treatments in nodular iron. The individual inoculants were chosen based on their specific chemical composition. By determining the effects of varying levels of specific active elements in the inoculants on the fading of magnesium when holding low production levels of molten nodular iron, better process controls can be implemented. Restoration of nodularity by means of a simple ladle addition permits improved productivity at a reduced cost, while maintaining or improving the quality of the metal poured. Results were determined on the evaluation of the microstructure, residual magnesium level, and Brinell hardness. Actual testing did not incorporate the LS VAC process.

Statement of the Problem

The problem of this research was to determine the influence of three specific, commercially available, inoculants on the fading effects of magnesium and inoculation during extended holding of molten nodular iron. The research interest was to investigate the application of casting thin-walled parts in ductile iron with the LS VAC process. The test samples included a 12.7 mm (0.50 in.) diameter and a 25.4 mm (1.00 in.) diameter cylindrical casting. This was done in an effort to evaluate the microstructure in the test castings with solidification rates similar to a 6.35 mm (0.25 in.) and a 12.7 mm (0.50 in.) thick plate. According to Angus (1976), it is a useful approximation to assume that the center of a flat plate will cool at the same rate as a round bar whose diameter is twice the thickness of the plate. The study compared the effects of the individual inoculants on the microstructural and mechanical properties of test samples taken over a period of 25 minutes.

CHAPTER II

LITERATURE REVIEW

Inoculation

Although the magnesium treatment is responsible for the development of spheroidal graphite, quality ductile iron also requires the use of an inoculant. Inoculation, or post-inoculation, refers to the practice of making an addition of elements to the melt that promote graphitization during solidification.

Purpose of Inoculation

Inoculants are believed to function by causing localized precipitation of graphite by forming nucleation sites. Inoculation is more essential to the production of ductile iron than gray iron for several important reasons. Uninoculated, pure magnesium-treated iron is almost always completely carbidic because the addition of magnesium in the ductile iron treatment process promotes undercooling which results in iron carbide (Fe_3C) formation as shown in Figure 1. Properly inoculated ductile iron contains no primary iron carbide in the microstructure, as shown in Figure 2. In addition to this, inoculation is critical in ductile iron because of the differences in solidification mode between gray and ductile iron.

Figure 1. Uninoculated ductile iron; 250X, etched.

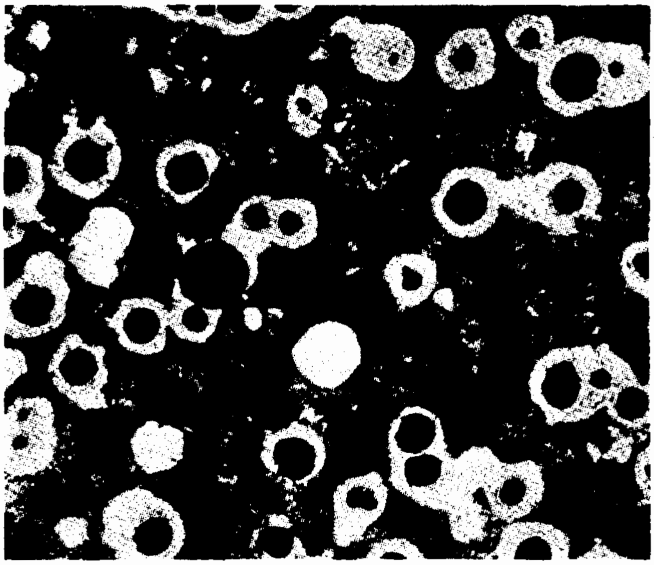

Figure 2. Properly inoculated ductile iron; 250X, etched.

In gray iron, graphite flakes grow in contact with the liquid, but in ductile iron the graphite crystals grow surrounded by a solid phase, the austenite shell. The relatively slow diffusion rate of carbon through the austenite shell requires more graphite nucleation sites to reduce the distance that carbon must diffuse, so that graphitization is achieved during solidification. According to Heine, Loper, and Rosenthal (1967) a greater number of graphite spheroids provides an increased number of sites for graphitization, thereby increasing the graphitizing tendency of the solidifying melt or reducing the chilling and shrinkage tendency. Without proper nucleation, carbides can be present in the as-cast ductile iron castings. This will result in castings with inferior ductility and machineability properties. Furthermore, the graphite formed from subsequent decomposition of the carbides may be irregular in shape, giving the appearance of iron treated with insufficient nodularizing alloy to provide graphite in the spheroidal shape. In general, the greater the degree of nucleation in ductile cast iron, the greater the number, and the smaller and more uniform in size and shape will be the graphite spheroids. According to Patterson (1975), "post inoculation is a key to structural control in ductile cast iron" (p. 141).

Method of Inoculation

The inoculation is the final step in the ductile iron production process. The addition of a small amount of an inoculant to the molten iron produces heterogeneous nuclei for the graphite spheroids to grow upon. The general advice is to treat first and inoculate afterwards. Inoculation effect fades with time and elevated temperature;

therefore, inoculating later in the metal transfer process is the traditional practice. However, according to a study by Campomanes and Goller (1973) the inoculant was added together with the magnesium treatment, and the results showed very little difference when comparing nodule counts with fading time. Additional advantages presented by their research included increased magnesium recovery; reduced magnesium flare, vapor and splashing during treatment; and lower temperatures required for treatment.

The one-step magnesium treatment and inoculation process, outlined by the Campomanes and Goller (1973) study, was utilized in the design of this research. This was due in part because of the low volume production being evaluated and also due to the low pouring weight of the melt. Due to the time restrictions imposed by these variables it was determined that ladle inoculation was an unsuitable option. Also, in-stream mold inoculation was impossible due to the pouring method being utilized. For the LS Vac process, the in-stream mold inoculation process is not feasible at this time, therefore, the one-step process was the best option available for the research.

Materials for Inoculation

Nearly all materials used as inoculants are based on some form of ferrosilicon (FeSi). According to a publication by the American Foundrymen's Society (1986), FeSi itself is not an effective inoculant, but the relatively small amounts of such elements as calcium, aluminum, strontium, barium, bismuth, and rare earths are thought to be the active agents responsible for the observed results of inoculation. Most likely others exist, the common characteristic of active inoculating elements is that they all form very stable oxides or, in other terms are strong deoxidizers.

The primary active elements of interest for this study included calcium and barium. After an extensive review of literature, only limited information was found concerning these elements and their effects as inoculating agents. Most of the studies were limited to low alloying levels of these elements.

Most of the graphitizing inoculants contain some calcium and aluminum. These elements are especially important in the ferrosilicon inoculants as they are more effective than ones not containing either element or ones containing just one element. Both calcium and aluminum are effective as nucleating agents in the ferrosilicon inoculants, provided the proper contents are maintained. However, aluminum increases the affinity for hydrogen pickup and thus pinhole porosity, therefore, the aluminum content should be kept low. Calcium is very effective as a deoxidizer and in reducing chill. In a study by Patterson (1973), it was stated, although not definitely proven, that at inoculating temperatures in excess of 1455°C (2651°F), higher calcium levels actually increased the holding power of the inoculant. However, high calcium levels can cause excessive slag with the possibility of slag entrapment in the castings.

The use of barium and its effect as an active element in inoculants has not been widely reported. From the review of literature, the previous study of barium has been limited to low addition levels, primarily in nodularizing alloys. According to Spengler (1984), barium in amounts from 2-2.5% were added to magnesium ferrosilicon alloys to promote a high nodule count in ductile iron and minimize the formation of centerline carbides in thin section castings. In the European and Asian research communities, it has been reported, according to Spengler (1984), that barium was used in amounts approaching 6.0% to a group of magnesium ferrosilicon alloys. According to these reports, these alloys could be used to treat base iron without the necessity of post-inoculation with ferrosilicon or other inoculants.

As an active element in inoculating alloys, barium functions in a manner similar to calcium in that it is a strong nucleating agent. However, barium has been reported by Patterson (1973), to make the inoculant effective over a wider temperature range than an alloy with calcium alone. Barium is beneficial as a nucleating agent when added to cast iron at temperatures in excess of 1480°C (2696°F). At temperatures ranging from 1370°C (2498°F) to 1430°C (2606°F), however, calcium seems to be slightly more effective than barium. Thus, the combination of barium and calcium in an inoculant makes it more effective over a wider temperature range. It was also reported by Patterson (1973) that barium appears to improve the holding power of the inoculant.

The utilization of high levels of barium containing inoculants has not been widely studied or reported. However, increased graphite counts in thin sections and improved mechanical properties of as-cast ductile iron was reported in a Russian study by Bulaevskii, Lerner, Senkevich, and Shitsman (1988). In their study, a high barium (15%) containing ferrosilicon alloy was added in amounts up to 0.9% to a 1% ferrosilicon post-inoculation treatment. In permanent mold casting of 5 mm (0.20 in.) thick samples, they reported that the addition of the ferrosiliconbarium lowered the overall carbide content from 55 to 20%, and simultaneously increased the ferrite content of the matrix.

Fading of Inoculation Effect

According to Janowak and Loper (1971), decreased nodule counts in nodular iron are a result of reduced post-inoculation effectiveness and are responsible for increased eutectic carbide formation. However, it is believed that nodularity fade and the fade of the post-inoculation effect occur independently. Independent fading effects, however, do not imply the absence on an interaction effect.

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. According to the literature reviewed, many factors influence the fading behavior of inoculants: section thickness, carbon equivalent (CE) and temperature.

Magnesium Fade

Magnesium fade, or fading of nodularization in ductile irons, is considered to be the loss of nodularity with time. Fading may occur due to the loss of magnesium from the melt as a result of its volatility, or due to reaction of magnesium with oxygen and/or sulfur. Fading may also occur due to a reduction in nodule count attributable to treatment and not to a change in magnesium content. According to Janowak and Loper (1971), graphite nodularity has been observed to decrease when magnesium residuals decrease but the kinetics of magnesium fade and the relationship between magnesium and nodularity have not yet been established.

Effects of Magnesium Fade on Microstructure

It is reported by Janowak and Loper (1971) that excessive magnesium fade and post inoculation fade results in decreased nodularity, reduced nodule counts, and increased carbide contents. Nodularity fade in properly treated nodular iron is evidenced by the formation of nonspheroidal graphite. It was identified by Janowak and Loper (1971) that nodularity does not decrease significantly until a magnesium level of 0.03% (magnesium equivalent of 0.0224) is attained. The magnesium equivalent is defined as %magnesium - 3/4 %sulfur. Continued magnesium fade below this level resulted in vermicular graphite formation. As magnesium residual levels approached 0%, flake graphite appeared.

Process Variables Affecting Magnesium Fade

As mentioned before, magnesium fade or the decrease in residual magnesium during extended holding of molten nodular iron, is thought to occur as a result of either chemical reactions or direct vaporization. According to Heine and Loper (1966), they have observed magnesium vapor rising from molten nodular iron surfaces. Flinn and Trojan (1965) believe that magnesium losses occur by vaporization and/or reaction with silica (SiO_2) and other components in refractories and suggest that induction stirring increases magnesium loss rates.

Other possibilities that affect magnesium losses include magnesium oxidation at the melt surface or magnesium reaction with sulfur and oxygen in the iron. According to a report by Heine, Janowski, Loper, and Wang (1976), it is essential that low sulfur content base irons be used to delay the loss of nodularity. They determined that in those cases where the sulfur level was low initially, the fading process was retarded and graphite shape remained primarily spheroidal until extremely low magnesium levels were achieved. Vermicular graphite was then observed for only a short time prior to the structure fading to complete flake-shaped graphite. Robertson and Vontress (1967) found that magnesium fade increases with higher melt temperatures, high concentrations of magnesium in the iron and higher exposed surface area to volume ratio of the melt.

From these studies, it is evident that the primary process variables that affect the magnesium fade in ductile iron have been identified as: refractory lining of the holding furnace, melt agitation or stirring action of the holding furnace, atmosphere over the melt, exposed surface area to volume ratio of the melt, isothermal holding temperatures, sulfur and oxygen level of the melt, and magnesium concentrations of the iron.

CHAPTER III

DESIGN OF THE STUDY

Introduction

The main experimental objective of the research work was to evaluate the effects of the individual inoculants on the fading effects of magnesium and inoculation in ductile iron. It was not the intent of the research to explain the physical metallurgy of property improvement provided by the independent variables.

The effects of the test inoculants were determined based on the residual magnesium, percent nodularity, nodule count and the hardness of the test samples. These variables were established based on an extensive review of literature on magnesium and inoculation fade. All variables were analyzed according to standard industry test methods. Optical emission spectrographic analysis, carbon sulfur determination, microstructural image analysis and Brinell hardness testing were used to evaluate these properties.

Because of the limitations of in-house evaluation equipment, outside sources were used. Optical emission spectrographic analysis for the determination of residual amounts of magnesium, and image analysis for microstructural verification were conducted at the ISO 9001 certified metallurgy lab at the John Deere Foundry, Waterloo, Iowa. The Brinell hardness testing was also performed at the John Deere Foundry, Waterloo, in accordance with ASTM E 10 Brinell Hardness of Metallic Materials standard.

A factorially designed experiment was conducted to observe the effects of the inoculation treatment over time on the magnesium (Mg) loss rates in a 250 lb. melt charge. A total of 3 heats were made to evaluate the inoculants under investigation. Additional heats were made for verification as needed.

Production of the Test Material

An effort was made to produce a consistent base melt for each of the three experimental heats. This consistency was based on maintaining the carbon and silicon levels following magnesium treatment and inoculation within the following specified ranges:

3.60 - 3.80% C
2.40 - 2.60% Si

A thermal differential analysis procedure, Electro-Nite™ DataCast 2000 Quick Lab, was utilized during the melting process to help control the carbon and silicon levels, in the base iron, within the specified range of 3.7 - 3.8% C and 1.4 - 1.5% Si prior to magnesium and inoculation treatment. The final silicon level was calculated based on the silicon content of the magnesium treatment master alloy and the inoculant being used and verified with optical emission spectrographic analysis.

Charge Materials

Charge materials for the experimental heats consisted of commercially available high quality materials including Sorel™ pig iron, AISI 1010 steel punchings, and 75%

FeSi. A 250 lb. charge mix was calculated in the ratio given in Table 1. All efforts were made to keep tramp elements (elements not accounted for in the original experiments) to a minimum. All metallic materials were clean and free of oil, rust or other contaminants to help reduce their possible detrimental effects.

Table 1

Melt Charge Mix and Addition Rates for a 250# Heat

Description of Charge Material	Weight (lbs.)	% of Charge
Sorel F-1 Pig Iron	55.16	22.0
AISI 1010 Steel Punchings	100.00	40.0
Carbon Raiser	4.23	1.7
Ferro Silicon 75%	2.76	1.1
Ductile Iron Returns	89.00	35.6

Melting Equipment

The experimental heats were melted at the Metal Casting Center located in the Industrial Technology Center at the University of Northern Iowa in a Pillar™ 300 lb. transit box induction furnace with a 180 KW power supply. The furnace uses a magnesia-alumina crucible as a working lining with a dry vibrateable magnesia refractory to hold the crucible and provide a safety lining. The crucible is further held in place by a top cap consisting of 75% alumina plastic refractory. This furnace lining is best suited in limiting the magnesium fade rate as reported by Janowak and Loper (1971) in a study of process variables and their effects on magnesium and nodularity fade in molten nodular iron.

Melting Procedure

After charging, the furnace was powered to 10% of the rated electrical capacity for ten minutes to prevent thermal shock to the furnace lining. Power to the furnace was then raised to 95% of rated electrical capacity until sufficient liquid formed and space was available for additional charge material. Using the Electro-Nite™ DataCast 2000 Quick Lab, a chemical analysis of the base iron was determined after meltdown and a minimum temperature of 1454°C (2650°F) was obtained. Trim additions were then added to the melt as necessary to obtain the desired chemistry range.

Once the base melt was established, the melt was brought up to 1510°C (2750°F) and a chill button was poured. This allowed for spectrographic analysis of the base melt chemistry. A schematic of the chill button is shown in Figure 3.

Magnesium and Post-Inoculation Treatment

Immediately preceding tapping, the magnesium bearing FeSiMg master alloy, in the quantity of 2% of the charge weight was placed in the Sigmet™ treatment chamber. In addition to the magnesium treatment master alloy, a specified amount of the experimental post-inoculant was placed in the flow-through chamber. The amount of post-inoculant was based on 0.5% of the total charge weight. The post-inoculation additions and critical elements for each experimental trial is outlined in Table 2.

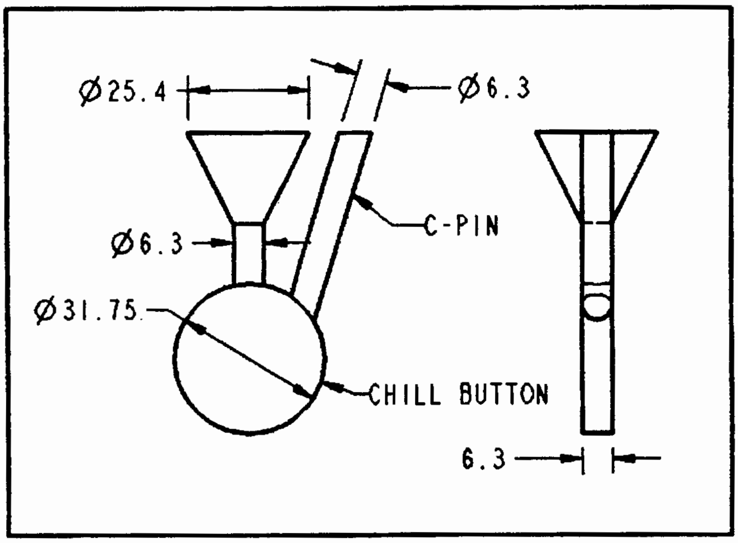

Figure 3. Schematic of the chill button produced for spectrographic analysis of the melt chemistry.

Table 2

Magnesium and Inoculation Treatment Addition Levels and Critical Element Compositions

Treatment Additions	Si	Ba	Ca	Mg	Weight (lbs.)	% of Charge
Inoculant A	75%	-----	-----	-----	1.25	0.5
Inoculant B	65%	5%	1.76%	-----	1.25	0.5
Inoculant C	57-62%	14-18%	14-17%	-----	1.25	0.5
Master Alloy	44.56%	0.81%	1.27%	6.32%	5	2%

Note. Not all elements are listed.

The base melt was then tapped into the Sigmet™ treatment chamber for magnesium and post-inoculation treatment and emptied into a 250 lb. KalTek™ pouring ladle. Figure 4 shows the Sigmet™ treatment chamber and the charging of the magnesium and post-inoculation treatment alloys. Immediately upon completion of the treatment process, the time and temperature of the melt were recorded. This assisted in determining the amount of heat lost during the treatment process. At that same time, a chill button was poured to determine the initial chemistry of the treated iron.

The 250 lb. pouring ladle used a KalTek™ shank as a working lining with a dry silica sand lining and a sodium silicate cap to hold the shank in place. Special efforts were employed to make sure that the Sigmet™ chamber and pouring ladle were clean of any previous melt residue to reduce the possibility of contamination from previous experiments.

The melt was then poured back into the furnace and adjusted to a temperature of 1345°C (2450°F). The temperature was then controlled at this level throughout the remainder of the procedure. Since there was no temperature feedback control for the induction furnace, periodic temperature measurements were conducted to monitor temperature variation. A variance of ± 20°C (±35°F) was obtained and could be maintained without difficulty.

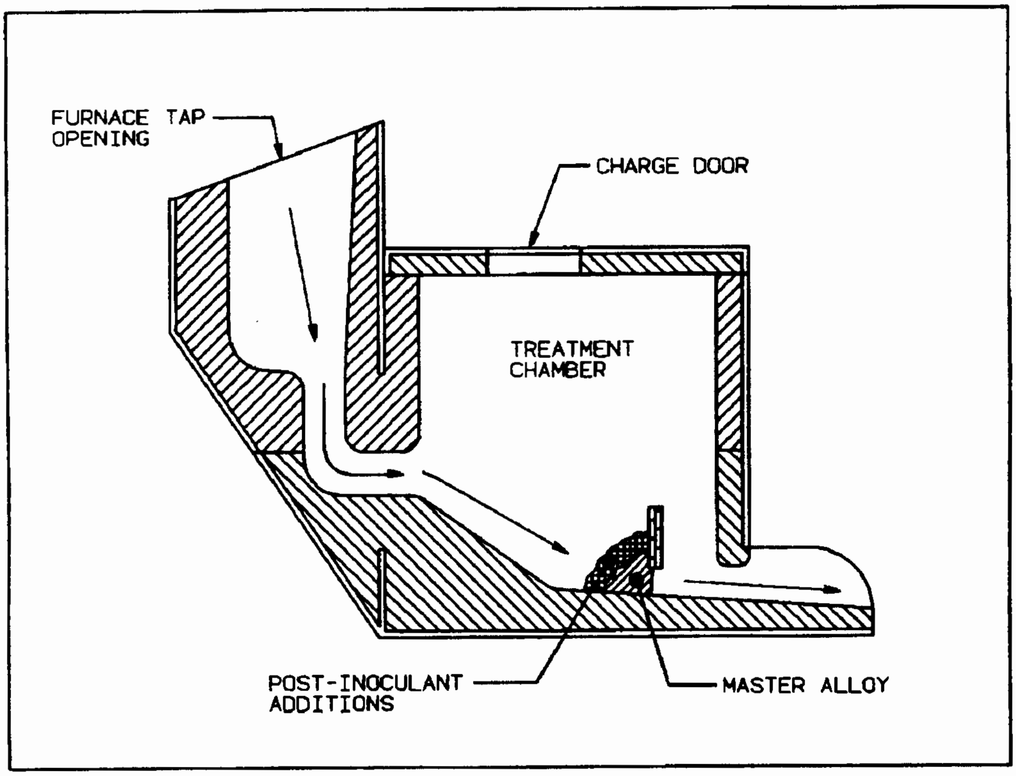

Figure 4. Schematic of the Sigmet™ treatment chamber and charging additions.

Experimental Procedure

An experimental procedure was established and followed for all experimental trials. An effort was made to incorporate the same sample procedures in all trials to maintain consistency and reproducibility.

Five minutes after the initial chill button was poured, the time and temperature were recorded, and an additional chill button and a set of test samples were poured in rapid succession. The test samples, including a 25.4 mm (1 in.) diameter x 76.2 mm (3 in.) long and a 12.7 mm (0.5 in.) diameter x 76.2 mm (3 in.) long cylindrical sample, were used for microstructural analysis. The molds for the test specimens were generated utilizing a phenolic-urethane binder process. This procedure was repeated every 5 minutes until 25 minutes had elapsed since the initial analysis was performed.

The test samples were then allowed to cool to room temperature before shakeout. Following shakeout, the samples were cleaned and stamped for identification and prepared for processing.

Analysis of Chemistry and Evaluation of Microstructural and Mechanical Properties

An extensive analysis of the chemistry, microstructure, and mechanical properties of ductile iron is the only means of determining and correlating the process variables that affect magnesium and inoculation fade during extended holding of molten nodular iron. Therefore, the analysis of the chemistry, microstructure and mechanical properties will be performed according to methods established through previous studies.

Residual Magnesium and Critical Element Determination

The chill buttons and C pins (used for carbon and sulfur determination) were used for the analysis of the test sample chemistry. The primary elements of interest were magnesium, carbon, silicon, sulfur, manganese, phosphorus, and trace element contents. The residual magnesium content was determined through the analysis of the chill button produced during each test sample. Optical emission spectroscopy was not available for the determination of the critical elements, calcium and barium, in this research, because the instruments available did not have specific channels set up to evaluate these elements.

Microstructural Analysis

The test specimens were sectioned to provide one 12.7 mm (0.5 in.) long sample and one 25.4 mm (1 in.) long sample. Two test samples were removed from each cast specimen. The locations of the sections was determined to avoid areas where porosity or shrinkage might be present, as outlined in Figure 5.

Test sample A, surface 1, was used to evaluate the microstructural properties of the test specimen. Two mid-radius regions were evaluated (indicated by a dotted line in Fig. 5) and averaged with respect to percentage nodularity and nodule count.

The percentage nodularity and nodule count of the test specimens were determined through the use of an image analyzer. Samples were read at 100X and the nodularity was calculated based on nodules/total number of particles expressed as a percentage. The nodule count was defined as nodules/mm^2.

Mechanical Properties Evaluation

Evaluation of the mechanical properties of the test samples were limited to Brinell hardness testing. Test sample B, surface 2, was used to evaluate the hardness properties of the test specimen. The Brinell hardness tester was equipped with a 10 mm (0.39 in.) diameter ball indentor and utilized an applied force of 3000 kgf (6614 lb·ft.).

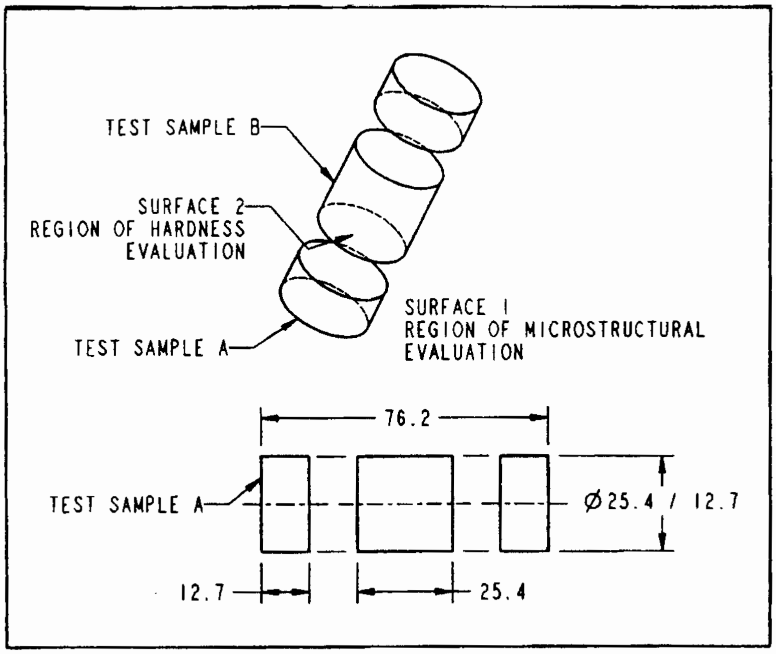

Figure 5. Schematic of test specimens and critical areas of test sample evaluation.

CHAPTER IV

RESULTS AND DISCUSSION

Evaluation of Chemical Analysis

The results were obtained to evaluate critical element values during extended holding of the molten nodular iron over the specified time of the experiments and to compare with the established specified ranges set forth prior to the experiments. The base melt ranges for carbon and silicon were established according to ductile iron standard melt operating practices for thin-wall castings. The study was designed to produce a hyper eutectic composition. The carbon equivalent (CE) specified range was established to be between 4.5 and 4.7%.

Evaluation of Temperature Control

To minimize the oxidation of magnesium from the melt, temperature control was critical following the treatment processes. All temperature data are presented in Table 3. The temperature control was maintained within the specified ranges established prior to experimentation. It should be noted that the temperatures were held below the effective nucleating temperature range of calcium and barium as reported by Patterson (1973). This was figured into the design of the experiments for several reasons. The temperature was to be minimized to limit vaporization and oxidation of the magnesium and to assimilate actual casting temperatures associated with the LS VAC casting process.

Table 3

Experimental Temperature (°C) Data

Sample	Time	Inoculant A Temp. (C)	Inoculant B Temp. (C)	Inoculant C Temp. (C)
Base Melt	NA	1516	1517	1513
Initial	00:00	1364	1406	1406
1	05:00	1332	1359	1346
2	10:00	1343	1360	1334
3	15:00	1351	1343	1336
4	20:00	1363	1352	1352
5	25:00	1353	1347	1357

Base Material Chemistry Evaluation

The results of the base melt chemistry for the individual experiments are given in Table 4. As can be seen, all element levels were consistent between experiments. It is important to point out the low levels of sulfur and manganese within the base melt. Sulfur levels were controlled to limit its reaction with magnesium during the treatment process. Manganese is a moderately strong carbide promoter, therefore its content was limited to avoid as-cast carbide formation. Maximum manganese levels are dependent on the silicon content and the section thickness of the casting.

Table 4

Chemical Analysis of Base Melt for each Experiment

Inoculant	C	S	Si	P	Mn	Ni	Cr	Mo	Cu	Sn	Ti	Mg
A	3.804	.0081	1.601	.011	.202	.056	.035	.003	.122	.009	.012	.001
B	3.719	.0137	1.725	.011	.211	.050	.034	.003	.116	.009	.013	.001
C	3.867	.0146	1.765	.012	.238	.049	.034	.003	.121	.010	.013	.002

Experimental Test Chemistry Evaluation

Chemical analysis of the experimental test samples were obtained to evaluate the residual magnesium and critical element levels of the test samples. The results of the chemical analysis for the test samples are given in Table 5. Slightly lower carbon levels were expected due to common carbon loss during magnesium treatment. The increased silicon levels can be accounted for due to the master alloy and inoculant additions. The initial levels of all three experimental inoculants were relatively consistent. The carbon equivalent (CE) values between the experiments were also maintained within the test range for all three experiments. Carbon and silicon levels were maintained within a respectable range, but more importantly, the residual magnesium level was established at $\geq 0.05\%$ for all three experiments. This provided an equal starting point from which to evaluate the three post inoculant treatments with respect to their fading rates.

Table 5

Chemical Analysis of Individual Trials for each Experimental Inoculant

Inoculant	Trial	C	S	Si	P	Mn	Ni	Cr	Mo	Cu	Sn	Ti	Mg	CE
A	Initial	3.525	.0072	2.882	.012	.200	.064	.029	.017	.127	.006	.006	.054	4.48
	1	3.727	.0051	2.861	.012	.199	.061	.025	.017	.128	.006	.006	.043	4.67
	2	3.644	.0049	2.844	.011	.194	.059	.025	.017	.115	.005	.006	.036	4.58
	3	3.696	.0031	2.931	.010	.204	.062	.025	.016	.121	.005	.006	.031	4.66
	4	3.627	.0035	2.885	.012	.205	.061	.025	.016	.127	.005	.005	.029	4.58
	5	3.703	.0032	2.882	.010	.197	.065	.026	.016	.113	.005	.003	.025	4.65
Inoculant	**Trial**	**C**	**S**	**Si**	**P**	**Mn**	**Ni**	**Cr**	**Mo**	**Cu**	**Sn**	**Ti**	**Mg**	**CE**
B	Initial	3.598	.0110	2.977	.011	.238	.049	.026	.005	.107	.005	.007	.054	4.58
	1	3.529	.0060	2.955	.011	.240	.053	.027	.006	.114	.005	.007	.040	4.50
	2	3.557	.0050	2.975	.010	.241	.055	.028	.007	.084	.003	.008	.033	4.54
	3	3.681	.0051	2.922	.011	.234	.044	.026	.005	.102	.005	.010	.032	4.65
	4	3.665	.0041	3.015	.011	.238	.047	.028	.005	.108	.005	.006	.026	4.66
	5	3.647	.0030	2.884	.011	.228	.045	.026	.005	.094	.004	.009	.024	4.60
Inoculant	**Trial**	**C**	**S**	**Si**	**P**	**Mn**	**Ni**	**Cr**	**Mo**	**Cu**	**Sn**	**Ti**	**Mg**	**CE**
C	Initial	3.524	.0124	2.803	.011	.240	.052	.029	.004	.112	.004	.008	.054	4.45
	1	3.548	.0121	2.724	.012	.236	.049	.027	.005	.114	.005	.004	.043	4.45
	2	3.686	.0073	2.760	.011	.242	.052	.030	.005	.112	.004	.008	.036	4.60
	3	3.569	.0049	2.732	.011	.240	.050	.027	.004	.015	.001	.000	.027	4.47
	4	3.600	.0032	2.816	.011	.239	.049	.028	.004	.114	.005	.005	.024	4.53
	5	3.702	.0034	2.709	.012	.231	.044	.026	.004	.104	.005	.005	.021	4.60

Microstructural Evaluation of Test Materials

This study utilized a 25.4 mm (1.00 in.) diameter test sample and a 12.7 mm (0.50 in.) diameter test sample to evaluate the microstructure and properties of the ductile iron. These samples were used to determine the post inoculants effect on magnesium fading and inoculation fading, or chill reducing tendency. The chill buttons were used for the evaluation of residual magnesium during the holding of the molten nodular iron.

Magnesium Fading Effect

Magnesium fading effect was evaluated based on residual magnesium content, percentage nodularity, and nodule count. The brinell hardness readings were evaluated to reinforce the effect of nodule count fade. Results of all the experiments for the 25.4 mm (1.00 in.) diameter test samples are given in Table 6.

Table 6

Data of Structure and Properties of 25.4 mm (1.00 in.) Diameter Test Samples

Inoculant	Test Specimen (trial)	Residual Magnesium (% Mg)	Percentage Nodularity (%)	Nodule Count (nodule/mm^2)	Brinell Hardness (BHN)
A	Initial	0.054	----	----	----
	1	0.043	90	178	201
	2	0.036	85	150	197
	3	0.031	65	142	193
	4	0.029	55	128	180
	5	0.025	30	106	130
B	Initial	0.054	----	----	----
	1	0.040	90	195	205
	2	0.033	85	193	189
	3	0.032	80	183	186
	4	0.026	60	126	178
	5	0.024	30	118	164
C	Initial	0.054	----	----	----
	1	0.043	90	195	195
	2	0.036	75	185	188
	3	0.027	70	173	180
	4	0.024	50	147	176
	5	0.021	30	133	157

The data clearly outlines, that for all experiments, the magnesium levels within the treatment batch decreased, or faded, with time. All experimental inoculants showed relatively similar and consistent decreases in residual magnesium levels, as displayed in Figure 6. From Figure 6, it can be seen that the fading rate of all experimental inoculants were similar, and after 25 minutes was in the range of 0.021-0.025% magnesium.

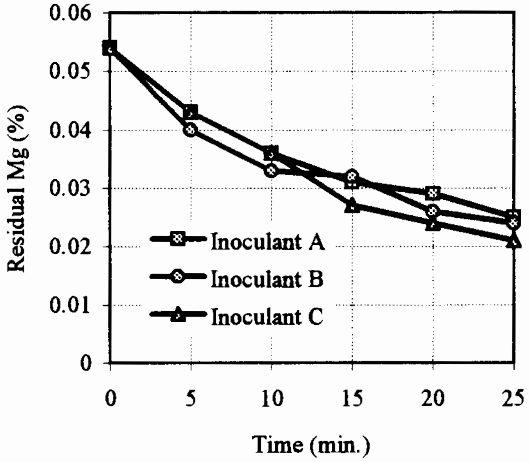

Figure 6. Decrease in residual magnesium levels for experimental inoculants over time.

It is also evident from Table 6, that as magnesium levels decreased, the percentage nodularity and nodule count also decreased with time. The decrease in percentage nodularity and nodule count for each experimental inoculant is also evident, as displayed in Figures 7 and 8, respectively. As can be seen in Figure 7, experimental inoculant B maintains a higher percentage nodularity over the first 20 minutes of the experiment. It should be noted that it is the only experimental inoculant that maintained at least 80%

nodularity for the first 15 minutes of the experiment. In Figure 8, it is evident that experimental inoculants B and C maintained a much higher nodule count during the first 15 minutes of the experiment. However, inoculant C demonstrated a more controlled and consistent trend in its nodular fade rate, over the duration of the experiment, than inoculants A and B.

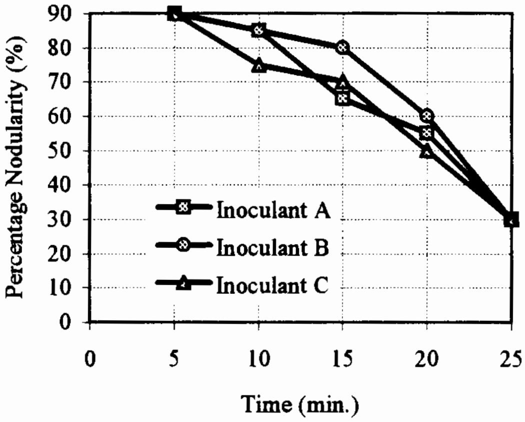

Figure 7. Decrease in the percentage nodularity for experimental inoculants over time, for 25.4 mm (1.00 in.) diameter samples.

The decrease in percentage nodularity and nodule count for each experimental inoculant is also evident in the photomicrographs presented in Figure 9. Five minutes after the magnesium and inoculation treatments, all three experimental inoculants show good uniform nodularity and nodule counts of nearly 200 nodules/mm^2 (as shown in Figures 9(a), 9(b), and 9(c)). Type A flake-graphite is present in all three experiments (as shown in Figures 9(g), 9(h), and 9(i)) within the first 15 minutes after treatment. Within 25 minutes after treatment, all three experimental samples are predominantly Type A flake-graphite with very poor nodularity and low nodule counts (as shown in Figures 9(m), 9(n), and 9(o)). The data indicates that only experimental inoculant B was effective in exceeding 80% nodularity while maintaining a high nodule count of 183 nodules/mm^2 for at least 15 minutes after treatment. The data also shows that 15 minutes after treatment, inoculant B had a slightly higher residual magnesium content than inoculants A and C.

The percentage nodularity for the 12.7 mm (0.50 in.) diameter test samples were observed to be about 80%, up to 15 minutes after treatment in all experimental tests (as shown in Figures 10(d), 10(e), and 10(f)). Experimental inoculant C demonstrated good graphite spheroidization while limiting carbide formation for in excess of 15 minutes, as shown in Figure 10(f).

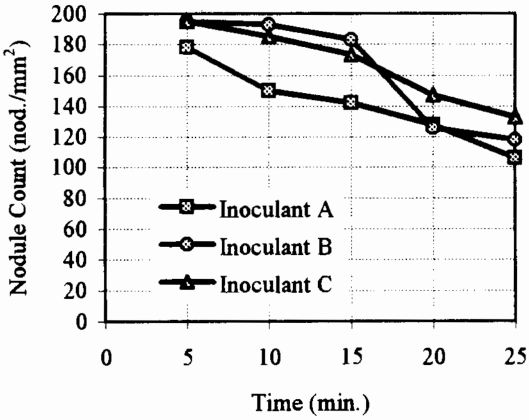

Figure 8. Decrease in the nodule count for experimental inoculants over time, for 25.4 mm (1.00 in.) diameter samples.

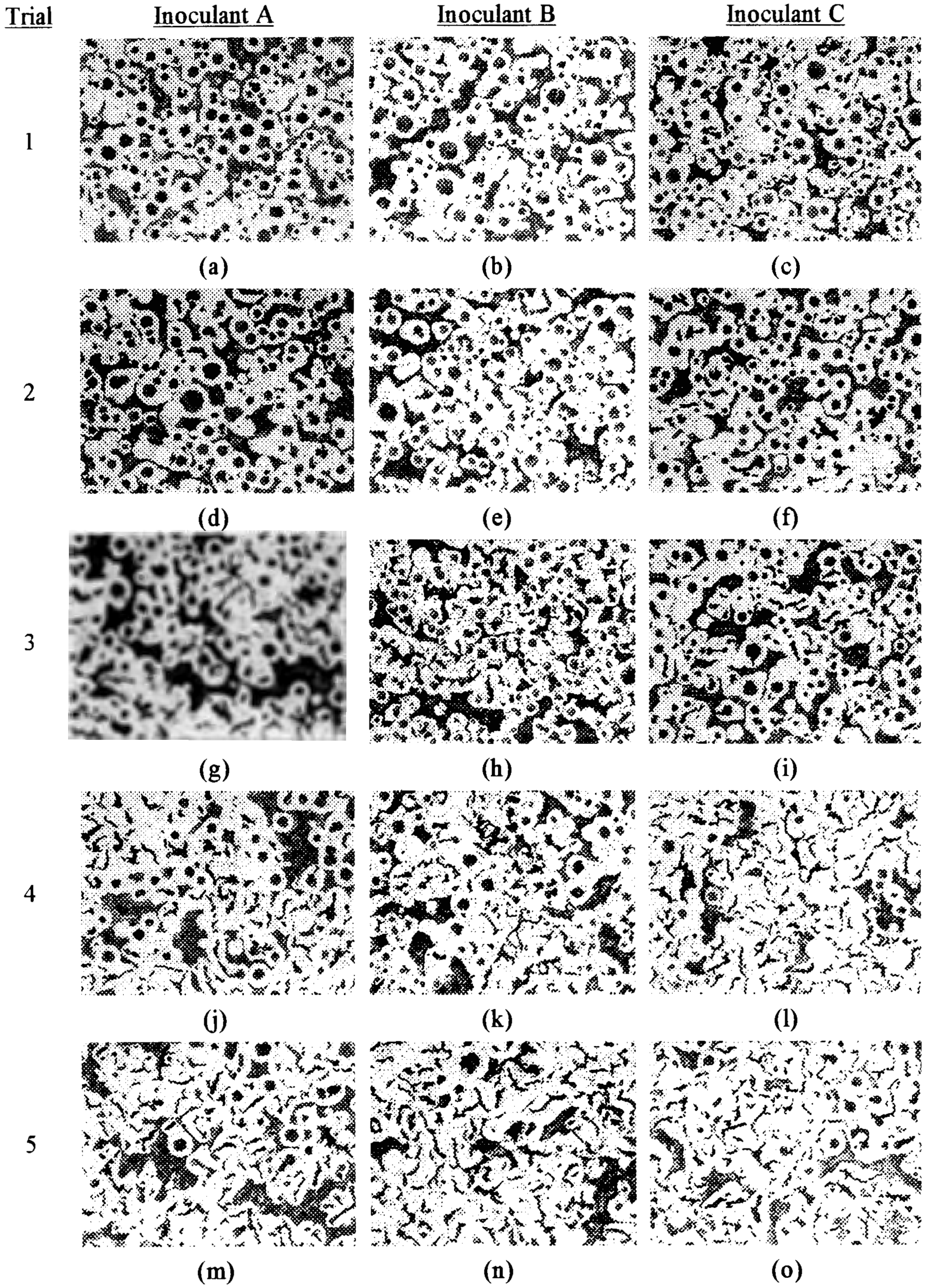

Figure 9. Photomicrographs taken at 100X, etched with a 4% Nital solution.

Inoculation Fading Effects

The 25.4 mm (1.00 in.) and 12.7 mm (0.50 in.) diameter samples were evaluated for iron carbide (Fe_3C) formation in an effort to determine the chill reducing tendency, or the resistance to inoculation fade, of the three experimental post inoculants. Figure 10 presents a series of photomicrographs taken from the 12.7 mm (0.50 in.) diameter test samples. For evaluation purposes, only trials 2, 3, and 4 were photographed. This represents test samples taken 10, 15, and 20 minutes after treatment.

It is evident from the photomicrographs shown in Figure 10 that the formation of free carbides was evident in all three experiments. However, the rate of free carbide formation was not consistent between the three experimental inoculants. Within the first 10 minutes after treatment, carbide formation is only evident within experimental inoculant A, as shown in Figure 10(a). The amount of carbides within this test sample would probably be considered acceptable, approximately 2-4%, but it should be noted that within 10 minutes after treatment, carbide formation had begun.

Within 15 minutes after treatment, both experimental inoculants A and B showed unacceptable levels of free carbides, in excess of 4%, as shown in Figures 10(d) and 10(e). Therefore, with inoculants A and B, the resistance to inoculation fade would be considered to be somewhere between 10 and 15 minutes. There is evidence of carbide formation within 15 minutes after treatment with experimental inoculant C, as shown in Figure 10(f), however, the amount of carbides present would be considered to be within acceptable levels. Unacceptable levels of free carbides do appear between 15 and 20 minutes after treatment with experimental inoculant C, as shown in Figure 10(i). It is evident, as shown in Figure 10, that experimental inoculant C offers more chill reducing tendency, or more resistance to inoculation fade than experimental inoculants A and B.

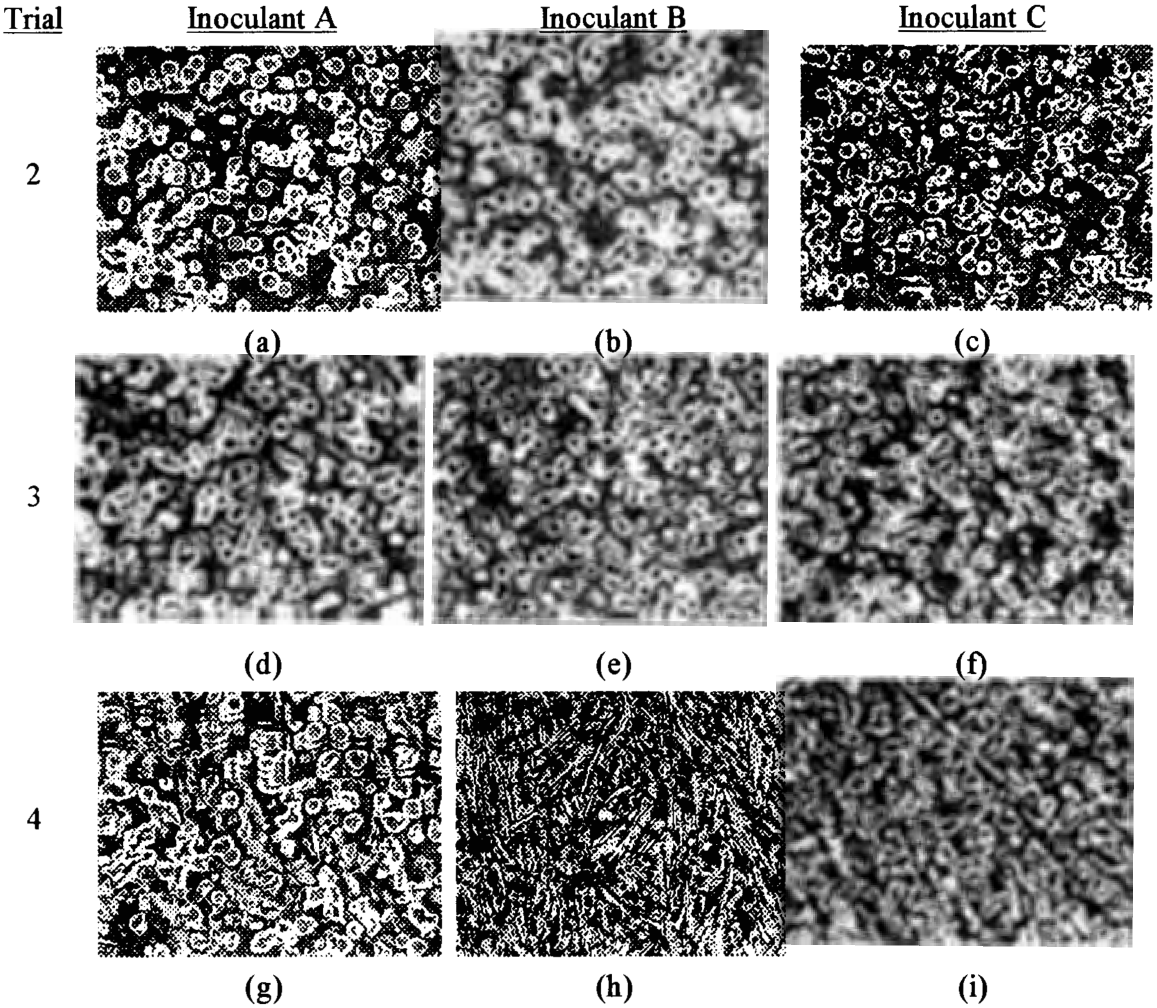

Figure 10. Photomicrographs taken at 100X, etched with a 4% Nital solution.

CHAPTER V

SUMMARY, CONCLUSIONS, AND RECOMMENDATIONS

This study involved the investigation of three specific, commercially available, post inoculants for ductile iron and their effect on the magnesium fading rate and chill reducing tendency in a low volume production environment. The one-step magnesium and inoculation treatment process was utilized in this study. The experimental inoculants used for the study were chosen based on their specific chemical composition. Experimental inoculant A, a foundry grade ferrosilicon 75% alloy, was chosen as the control group. Experimental inoculants B and C were chosen because of their varying levels of calcium and barium in the alloy. Inoculant B contained a moderate level of barium within the alloy, while inoculant C contained high levels of calcium and barium within the alloy. Although the review of literature reported that calcium and barium act similarly as strong nucleating agents and strong deoxidizers, only one study was found that reported their use as post inoculants in high alloying levels. The results of that study were favorable, however it was used in a two-step treatment process and in conjunction with a foundry grade ferrosilicon 75% inoculant addition, and was limited to permanent mold castings.

The research focused on two areas, magnesium and inoculation fading effects. Results obtained from the three experiments regarding any beneficial effect on magnesium fade, showed that there was no substantial differences between the three experimental inoculants. After holding the nodular iron for 25 minutes, all three experimental inoculants showed residual magnesium levels within the range of 0.021 - 0.025%. The residual magnesium levels, percentage nodularity and brinell hardness values all decreased with time at similar rates. The data collected through image analysis did show that the experimental inoculants B and C had slightly better nodule count values. It is evident that the experimental inoculants B and C held the nodule count better during the first 15 minutes after magnesium and inoculation treatment. However, there was a sharp decrease in the nodule count between 15 and 20 minutes after treatment.

There were some positive effects demonstrated by the experimental inoculants B and C in terms of their chill reducing tendency in the 12.7 mm (0.5 in.) diameter test specimens. Inoculant A, the foundry grade ferrosilicon 75%, showed carbide formation within 10 minutes after treatment, while inoculant B, a moderate level barium containing alloy limited carbide formation for 10 to 15 minutes after treatment. The high barium and high calcium containing inoculant C, proved to be the most effective chill reducer by limiting carbide formation in excess of 15 minutes, while maintaining an acceptable nodularity of about 80%. Experimental inoculant C may be recommended for thin-wall castings produced using countergravity processes as well as other molding methods.

Recommendations for Future Research

Due to the constraints of resources and equipment placed on the research it would be recommended that future studies include an investigation into the oxygen, calcium, and barium levels during the holding of the nodular iron. This study was unable to determine and track calcium and barium levels quantitatively. This would be beneficial in understanding the role of these deoxidizers. In addition, tracking oxygen levels of the molten iron in the holding furnace could be beneficial in understanding and controlling the calcium and barium levels.

Further research should be preempted with a better understanding of the recovery rates of the master alloy and post inoculation additions utilized in the one-step treatment method within the Sigmet™ treatment process. Also, studies should be developed in formulating customized addition rates of the post inoculants to best utilize the deoxidizers within the alloying additions.

Because of the process concerns associated with holding nodular iron in an induction furnace, process variables need to be carefully addressed. Temperature control variability needs to be controlled to minimize temperature fluctuations and excessive induction stirring of the melt. Another process option to help extend the fading rate would be to incorporate an inert, or neutral, atmosphere over the melt to minimize oxidation of the magnesium from the melt.

REFERENCES

Angus, H. T. (1976). *Cast iron: Physical and engineering properties* (2nd ed.). Boston: Butterworth Publishers Inc.

Bulaevskii, Y. V., Lerner, Y. S., Senkevich, Y. I., & Shitsman, E. B. (1988). Graphitization of iron with barium-containing additives. *Liteinoe Proizvodstvo, 9*, 76-77.

Campomanes, E., & Goller, R. (1973). One-step treatment for ductile iron. *Transactions of the American Foundrymen's Society,* pp. 428-432.

Ductile iron: Molten metal processing (2nd ed.). (1986). Des Plaines, IL: American Foundrymen's Society.

Flinn, R. A., & Trojan, P. K. (1965). Fundamentals of magnesium addition to ductile iron. *SAE Transactions,* pp. 265, 294.

Heine, R. W., Janowski, L., Loper, C. R., Jr., & Wang, C. C. (1976). Fading of magnesium treatment in ductile cast irons. *Transactions of the American Foundrymen's Society,* pp. 203-214.

Heine, R. W., & Loper, C. R., Jr. (1966). Dross formation in the processing of ductile iron. *Transactions of the American Foundrymen's Society,* p. 274.

Heine, R. W., Loper, C. R., Jr., & Rosenthal, P. C. (1967). *Principles of metal casting* (2nd ed.). New York: McGraw-Hill.

Janowak, J. F., & Loper, C. R., Jr. (1971). Process variable effects on magnesium and nodularity fade in molten nodular iron. *Transactions of the American Foundrymen's Society,* 594-599.

Patterson, V. H. (1973). Inoculants for grey and spheroidal-graphite iron: Their use and effect. *Foundry Trade Journal,* 91-104.

Patterson, V. H. (1975, October). Post inoculation of ductile iron. *Proceedings of Joint American Foundrymen's Society - Ductile Iron Society,* Rosemont, IL

QIT-Fer et Titane Inc. (1992). *Ductile iron production I: The state of the art, 1992,* (rev. ed.). Canada: Karsay, S. I.

Robertson, I. C., & Vontress, W. R. (1967, July). Using unalloyed magnesium in ductile iron production. Foundry, p. 74.

Spengler, A. F. (Ed.). (1984). The ductile iron process. (Available from Miller and Company, 55 East Monroe St., Chicago, IL 60603)