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EFFECTS AND LIMITATIONS OF RESIDUAL ALLOYING ELEMENTS IN SILICON SOLID SOLUTION STRENGTHENED FERRITIC DUCTILE IRON

Julia Scruton

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Scruton, Julia, "EFFECTS AND LIMITATIONS OF RESIDUAL ALLOYING ELEMENTS IN SILICON SOLID SOLUTION STRENGTHENED FERRITIC DUCTILE IRON", Open Access Master's Thesis, Michigan Technological University, 2018.

<https://doi.org/10.37099/mtu.dc.etdr/748>

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EFFECTS AND LIMITATIONS OF RESIDUAL ALLOYING ELEMENTS IN
SILICON SOLID SOLUTION STRENGTHENED FERRITIC DUCTILE IRON

By

Julia M. Scruton

A THESIS

Submitted in partial fulfillment of the requirements for the degree of

MASTER OF SCIENCE

In Materials Science and Engineering

MICHIGAN TECHNOLOGICAL UNIVERSITY

2018

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This thesis has been approved in partial fulfillment of the requirements for the Degree of
MASTER OF SCIENCE in Materials Science and Engineering.

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Acknowledgements

I would like to thank the Ductile Iron Society especially the research committee and Jim Wood for the funding of this project.

Dr. Paul Sanders who served as my advisor and provided a lot of guidance and feedback as well as helping to keep me on track.

Dale Dewald and Matt Otte who served as foundry managers and helped me to prepare and execute the twenty-two pours.

Lizeth Medina from Neenah Foundry who encouraged me to submit the project proposal and provided chemical analysis and constructive feedback.

Alex Basaj who ran green sand and helped tend the furnace for most of my pours.

Alejandra Almanza who assisted with the pours, analysis, and was a wonderful officemate.

Russ Stein, Paul, Fraley, and Dan Sequin who assisted with sample preparation and material testing.

The many colleagues who helped with pours: Sascha Becker, Lauren Bushong, Sam Dlugoss, Matt Makarewicz, Nick Martin, Chris Pflug, Phil Staublin, Riley Stimpson, Carson Williams, and Tom Wood

Abstract

Transitioning from traditional grades of ductile iron to solution strengthened ferritic ductile iron (SSFDI) grades can be difficult because of the need to reduce the amount of pearlite promoting elements, particularly copper and manganese, which are used in traditional grades. Knowing the acceptable threshold of residual alloying elements will allow for more cost-effective tailoring of the charge stream. Data were generated from a series of casting trials with the target grade of EN-GJS-500-14 where the levels of copper and manganese were varied from 0.05% to 0.3% and 0.15% to 0.4% respectively while maintaining silicon in the 3.8%-4.3% range. The pearlite content and tensile properties were measured and analyzed. Using this data, a relationship connecting the copper, manganese, and silicon content in the iron to the pearlite content was developed and the effect of these high residuals on tensile properties was quantified.

1 Introduction

1.1 Ductile Iron

Ductile iron is an engineering material composed primarily of carbon, silicon, and iron. Cast iron differs from steel in that cast iron has free graphite in an iron matrix. Ductile iron (DI) or spheroidal graphite iron contains graphite in the form of nodules or spheroids, this “ductile” material was discovered in 1948 [1]. The most commercially common treatment is done with magnesium additions. Cast irons are viewed as preferable to steels because the composition is nearly eutectic and as the melting temperature is typically 350°C less than that of steel it requires less energy to melt. Another reason that cast iron is preferred to steel is that the presence of the free graphite works to counter the shrinkage that occurs as the material solidifies resulting in less shrinkage, smaller risers, and better casting yields (weight of metal shipped divided by weight of metal poured) than steel. The mechanical properties of ductile iron are superior to grey iron or lamellar graphite iron because the graphite morphology of spheroids does not cause the stress concentrations and micro cracks that are in grey iron [2].

1.2 Ductile Iron Grades

Ductile iron grades are based not on chemistry like many alloy systems, but on expected mechanical properties [3]. The iron grades are specified by Ultimate Tensile Strength (UTS), yield strength, and elongation to failure. The European system only uses UTS-%El in the grade name (Table 1.1). A property-based specification system provides freedom so that the composition and processing can be tailored to the specific part geometry or the chemistry can be designed to be as cost effective as possible.

Table 1.1 European Ductile Iron Grades (with typical yield strength)

Grade	UTS (MPa)	Yield (MPa)	Elongation (%)
EN-GJS-400-15	400	250	15
EN-GJS-450-10	450	310	10
EN-GJS-500-7	500	320	7
EN-GJS-600-3	600	370	3

1.3 Alloying Elements

Alloying elements in ductile iron play a major role in the casting’s final properties. Carbon and silicon are two major influences on solidification behavior, magnesium promotes the spheroidal graphite morphology, and copper and manganese influence the amount of pearlite present [4].

1.3.1 Carbon Equivalent

The carbon equivalent (CE) indicates the iron composition relative to the eutectic. The silicon and carbon are combined into the carbon equivalent on the composition line of the phase diagram and the CE eutectic composition is about 4.3% CE. The simplest form of the CE equation only considers carbon and silicon composition (Eq. 1). Silicon stabilizes the graphite phase by lowering the metastable cementite (Fe_3C) formation temperature [5].

$$CE = C + \frac{1}{3}Si \quad (1)$$

1.4 Traditional Ductile Iron

Traditional ductile iron has a mixed matrix composed of ferrite and pearlite surrounding the nodules, with the strength increasing with pearlite fraction. These iron grades typically have a 3-4% carbon and 1.8-2.8% silicon, using these two elements to target a slightly hypereutectic CE (e.g. 4.5%) [6]. Other intentionally added elements in traditional grades are magnesium, copper, manganese, nickel, tin, molybdenum, antimony, barium, and some rare earth elements. Elements generally viewed as contaminants are sulfur, phosphorus, and some tramp elements from alloyed scrap steel used in the charge [4].

The microstructure of traditional ductile iron grades often has shells of ferrite around the nodules, called bullseye ferrite, as the carbon is able to diffuse out of the austenite and into the nodules. Carbon in austenite far from a nodule is trapped, so the carbon-rich austenite transforms into the pearlite composed of alternating layers of ferrite and the brittle intermetallic cementite (Fe_3C).

The ratio of the ferrite to pearlite in traditional ductile iron determines the mechanical properties. As the ratio of pearlite to ferrite increases the yield and tensile strengths increase, while the elongation and fracture toughness decreases [7]. For example, the 500-07 grade has approximately 30% pearlite, while the 700-02 grade has about 85% pearlite [7]. A difficulty with the mixed pearlite and ferrite ductile iron is that the amount of pearlite is influenced not only by the alloying additions and the overall chemistry but is also strongly tied to the section size. Brockus in his paper entitled *Effect of Section Size on Producing Ductile Iron Castings* illustrates this strong effect [8] as the pearlite was virtually 0% in 24 mm sections and 80% in 4 mm sections. Changes in pearlite fraction with section make it difficult to meet mechanical targets with complicated features and different section thicknesses. The alloy composition may be altered for mechanical properties depending on the part geometry.

1.5 Solution Strengthened Ferritic Ductile Iron (SSFDI)

Solid solution strengthened ferritic ductile iron is considered by some to be the second generation of ductile irons. Instead of using a pearlite-ferrite matrix to provide strength,

the solution strengthened ferritic ductile iron is strengthened by silicon in solid solution in a fully ferritic matrix. The matrix is fully ferritic due to the higher silicon level so the cooling rate and therefore the section size of a casting have less impact on the mechanical properties of the material [9].

A primary difference between solution strengthened ferritic ductile iron and traditional pearlite-ferrite irons is the hardness is more uniform. Due to the large difference in hardness between the pearlite and the ferrite in traditional irons it had localized hard and soft areas because of the changes in cooling rate at the different locations in the part as well as process variations. This is reflected in the international standard where they have a single grade of solid solution strengthened ferritic ductile iron of 500-10 with a specified hardness range of 185-215 Brinell [10]. The range for the 500-07 pearlite-ferrite iron is from 170-230 Brinell, which is twice as large as for the solution strengthened grade and overlaps with all of the other grades of traditional ductile cast iron [10].

The relationship between silicon content and mechanical strength was studied by Lacaze et al. where he determined that the strength of the alloys keeps rising up to 4% silicon [11]. Other studies have shown that the yield and ultimate tensile strength for the solid solution ferritic ductile irons increases as the amount of silicon increases up to about 4.3 percent then they start decreasing due to the embrittlement of the ferrite [12]. The hardness increases as more silicon is added due to ferrite solid solution strengthening [12]. At equivalent tensile strength in traditional ductile iron and solution strengthened ferritic ductile iron (up to 4.2 wt. % Si), the elongation in the ferritic iron is significantly higher. The ratio of the yield to tensile strength in the SSFDI is greater than that of the traditional mixed pearlite ferrite iron, with values of about 0.65 compared to 0.55 [12].

The effect of the high silicon is not just an increase in the strength and hardness. Higher silicon reduces carbides (including cementite in the pearlite) resulting in a nearly fully ferritic matrix in the absence of strong pearlite promoters. Less pearlite in the matrix leads to more matrix carbon diffusing to graphite, but the volume fraction of the graphite does not increase because at constant CE the high silicon levels decreases the overall carbon level (Eq. 1) [12]. Carbon diffusion distances are reduced due to smaller nodule spacing due to silicon being a strong graphite promoter [12].

Higher levels of silicon increases the ductile to brittle transition temperature and decreases the fracture toughness compared to fully ferritic ductile iron, but solution strengthened iron performs the same or slightly better when compared fracture toughness of the pearlite-ferrite iron of the same strength [13]. In heavy section ductile iron castings, higher silicon castings are more likely to form non-ideal graphite morphologies such as chunky or exploded graphite because the silicon reduces the austenite shell melting temperature allowing nodules to decompose. In addition, heterogeneous nucleation can lead to more chunky graphite and clustering of graphite nodules to form large masses of chunky graphite [11]. Excess chunky graphite decreases fracture toughness as poorly formed graphite initiates micro cracks [11].

1.6 Commercialization of SSFDI

Solution strengthened ferritic ductile iron production is increasing due to improved machinability, consistent mechanical properties, and light weighting potential. A case study presented by R. Larker in 2008 demonstrated the use of solution strengthened iron as a replacement to the 500-07 grade of pearlite-ferrite mixed iron for hydraulic rotors [13]. The 500-7 iron grade had to be annealed prior to machining to reduce the hardness fluctuations in the casting and part-to-part variation that caused difficulties in meeting the 20-micron machining tolerances [13]. When this part was switched from the traditional grade to the SSFDI grade the hardness variation was reduced from 40 HWB to 15 HWB [9].

A machinability study was conducted by Bjorkegren on SSFDI to investigate a possible reduction in hardness variation and improved machining [13]. This study saw significant reductions in hardness variation within the casting itself and a 6% improvement of machinability with a calculated potential for 10% reduction in machinability costs [13].

Lightweight applications are also possible as SSFDI is less sensitive to cooling rate allowing thinner sections to be cast without embrittlement from excessive carbides [7,9]. The increased yield to UTS ratio allows design engineers to utilize thinner parts as they design to a higher relative yield strength. Another benefit for lightweighting is even with no design changes this SSFDI alloys are slightly less dense than the traditional iron grades. An iron that was equivalent to a grade 500-07 has a density of 7.10 grams per cubic centimeter, whereas the 500-10 grade has a density of 7.04 grams per cubic centimeter. This is a difference of just under 1%, which is small but not insignificant [14].

1.7 Motivation

In SSFDI, pearlite fractions above 5% are unacceptable and it is known that Mn levels must be below 0.50 wt.% to lower the risk of exceeding the pearlite threshold (DIN EN 1563). Unfortunately, the combined levels of other pearlite promoting elements such as Cu, Ni, Cr, and Sn are not specified. These pearlite promoting elements are commonly found in more conventional ductile iron grades and therefore they may be incorporated into SSFDI heats from the heels of previous melts.

Due to uncertainty about the influence of pearlite formers, many foundries may choose to pig the heels of conventional iron grades before producing SSFDI compositions. This cost in raw materials, energy, and time may impede SSFDI implementation. Better understanding of the effects of pearlite promoting elements could enable foundries to more efficiently utilize their melting resources when casting SSFDI to realize manufacturing cost savings. Information on the pearlite promotion potency in SSFDI would also aid in lowering costs associated with charge make-up. Lower cost additions of scrap steel and returns could be used instead of higher cost virgin materials as long as the maximum thresholds for pearlite promoters are avoided.

1.8 Hypothesis

If the silicon content of an alloy is increased to enable solution strengthening in ferritic ductile iron, then the iron can tolerate increased copper and manganese contents before pearlite is formed as silicon will suppress carbide formation.

2 Experimental Procedure

2.1 Selection and Determination of Alloys and Charge Mix

Each charge was composed of pig iron, scrap steel, DI returns of tradition grades, SSFDI returns, and other alloying additions as needed. The charge composition was calculated using a mass balance spreadsheet to achieve the desired chemical compositions (appendix A).

Two sets of alloys were cast. Alloy Set A had 10 chemistries selected to provide a structured look at common residual alloy quantities for copper and manganese (Figure 2.1). The goal was to maintain silicon between 3.7 and 4.0%, with the carbon between 3.1 and 3.3%. This iron was inoculated with a 0.2% Topseed® addition to the ladle and a 0.1% by weight in-mold addition of Alinoc 67 (20x70 mesh size).

Figure 2.1. Target chemistries for Alloy Set A.

Alloy Set B had 5 compositions and covered an expanded range of alloying levels (Figure 2.1). An additional heat was added to assess the highest alloy content with less silicon. Set B was inoculated with Topseed as magnesium cover in the ladle and Spherix outside the pocket. These were the only inoculating steps, so the spectroscopy data is representative of all the castings. The mass balances for Alloy Set B are shown in Appendix A.

Figure 2.2. Alloy Set B target chemistries overlaid on Alloy Set A.

2.2 Mold Preparation

2.2.1 Mold Queue

2.2.1.1 Alloy Set A

Alloy Set A was cast into 5 vertically parted tensile bar molds. Each mold contained four blanks that could be easily turned into tensile bars. These blanks are 0.75 inches (19 mm) in diameter and 8 inches (200 mm) in height (Figure 2.3). These molds were made from an air set chemically bonded sand (Figure 2.4). Each mold half required 22 lbs. of sand, resulting in a total mold weight of 44 pounds of sand. The weight of these casting systems was 11 lbs.

Figure 2.3 Tensile Bar Blank Casting

Figure 2.4 Tensile bar mold

Two step bar molds were also cast for each heat. The bar has steps that were 0.25, 0.5, 0.75, and 2 inches high. This mold was made from green sand and was horizontally parted (Figure 2.5). This casting system weighed 12 lbs. Additional metal was poured into pig mold which can hold up to 120 lb of iron but typically were filled to between 60-80 lbs for ease of recharging.

Figure 2.5 Step bar casting used for section size and cooling rate analysis.

2.2.1.2 Alloy Set B

Alloy Set B was used to cast 5 tensile bar sets and 2 step bars. The tensile bar molds had a 55x55x12.7 mm filter with 2.31 mm diameter holes positioned at the bottom of the pouring cup (Figure 2.6). In addition, 3 y-blocks were cast (Figure 2.7) with a 23.5 mm cross section at the bottom. All three 18 lb y-blocks were cast in the same 106 lb chemically bonded mold (Figure 2.8). The y-blocks were open poured and had sand placed on the exposed top after approximately 6 minutes.

Figure 2.6 Tensile bar pattern with filter for 0.75 in diameter by 8 in tall cylinders.

Figure 2.7 Y-block schematic (in mm).

Figure 2.8 Y-block mold layout (in mm)

2.2.2 Chemically Bonded Sand

This chemically bonded sand was air set using an ALPHASET® 9010 resin system mixed in a Tinker Omega TOM. The total addition to the sand was 1.25% by weight with 30% catalyst and 70% resin. Molds were packed up to two weeks ahead of time for each pour with the typical time being 2-5 days.

The tensile bar molds were made in two mold boxes. After about 3 seconds sand with resin was ejected, so after an additional second the sand was directed into the mold box. The sand was well-packed by hand, then a bar was used to screed off the excess. The next mold box was packed, and they cured for approximately 12 minutes. For Alloy Set B a filter print was carved into both halves of the mold. The loose sand was then vacuumed out of the mold halves and the molds were assembled on the baseplates (filters were added for Alloy Set B) and clamped together in a set of 3 and a set of 2. The molds on the baseplates were then checked for leaks using a flashlight, then transferred to the pouring line where inoculant was added for Alloy Set A.

The y-block mold was made using a single mold that had 3 y-block patterns in it. This mold was vibrated using a vibrating table when the mold was approximately two thirds full and again after the program had ended. The excess sand was then removed using a screed. The mold sat for approximately 15 minutes before it was flipped onto a baseplate and the mold was tapped loose from the mold box.

2.2.3 Green Sand

The green sand molds were made from a silica sand that was activated by bentonite and water. The sand was approximately 4% bentonite by weight. Water was added, and the

sand was mulled until a compactability of between 35 and 50 percent was achieved. The molds were packed while the metal for each heat was melting to prevent the molds from drying out.

The step bar mold was made using a match plate and ring flask method. The match plate was placed in-between the cope and drag of the flask. The drag portion of the mold was packed first, and sand was riddled over the pattern until approximately a half inch of sand covered the whole pattern. Then approximately 2 inches of green sand was placed on top of the riddled sand and rammed against the pattern into the mold. A second 2 inch layer of greensand was added and packed down, and this was repeated until the flask was overfull. A metal screed was used to plane the drag. The whole flask was then flipped onto the base plate so that the newly packed drag was down. The cope then had sand riddled into it until the whole pattern was covered and it was packed in the same manner as the drag. The mold was screeded flat and the sprue was cut using a metal sprue cutter. A pouring cup approximately 3 inches in diameter and an inch deep was cut around the sprue to provide a larger target for pouring. The cope was then removed from the drag to remove the match plate. The molds halves were blown clean to removed potential sand inclusions and the flask was reassembled. The mold on the baseplate was then transferred to the pouring line rollers. Once it was on the line, a measured amount of in-mold inoculant was added (for Alloy Set A only), lake sand was used to mark around the pouring basin, and approximately 40 lbs of mold weight was placed on top of the mold.

2.3 Chemical Analysis

All chemistry samples from Alloy Set A were sent to Neenah Foundry for chemical analysis. The majority of the elements were measured by optical emission spectroscopy (OES), while combustion analysis was used for more accurate carbon and sulfur values.

Chemistry samples from Alloy Set B were tested in the Michigan Tech Foundry using a Bruker Q4 Tasman optical emission spectrometer. The chemistry samples were then sent to Neenah Foundry to verify the chemistry results.

For Alloy Set A the 0.1% addition of inoculant that was added to the molds were not accounted for in the OES button collected from the ladle. This silicon values was calculated based off of the addition rate and the ladle iron chemistry (Table 3.1)

2.4 Melting and Solidification

All 250 lb heats were melted in the Michigan Tech Foundry using a 300 lb capacity coreless induction furnace. The charge material was prepared by sand blasting the pig iron until most of the rust was removed and sand blasting or wire brushing to remove the excess sand from the SSFDI returns. The ferrosilicon, carbon, and ferromanganese additions were added to the furnace first, with the large returns from SSFDI heats added next, then ductile iron returns and pig iron were added until the furnace was full, and

finally steel punchings were poured over the top. Remaining ductile iron returns and pig iron was added as the iron melted down, and the copper additions (if necessary) were added after all the metal had melted. The iron was then heated to around 1420°C and for Alloy Set A and B a tellurium cooling cup was used to determine silicon and carbon content for the base iron and for later analysis two OES buttons were poured. For Alloy Set B an OES button was analyzed for silicon and other elements in addition to the data from the cooling cup. Alloying additions were made to the base iron to adjust chemistry as needed. When the base iron chemistry was deemed acceptable the iron temperature was raised to 1475°C, deslagged, and tapped.

The iron was treated with magnesium in the tundish ladle process. The magnesium ferrosilicon alloy was added to the pocket, barium containing Topseed® was added, and then it was all covered by 1040 steel punchings. After the magnesium reaction was finished an OES button and a cooling cup with sulfur and tellurium were poured for chemical analysis. Then the molds were poured; from taping of the furnace to emptying the ladle took less than six and a half minutes. The castings were removed from the molds after they were cool to be handled.

2.5 Sample Preparation

2.5.1 Tensile Bar Mold

The tensile bar samples were taken from the middle of tensile rod castings. The rods were angle ground to separate them from the runner. The round blanks were then cut to 6-inch lengths by removing approximately two inches from the top of the bar and then turned on the CNC lathe. Metallography samples were taken from the outside bar near the runner (Figure 2.9).

Figure 2.9 Location of tensile sample from tensile bar blanks (arrow indicates the surface analyzed).

2.5.2 Step Bar Mold

The step bar was sectioned along each step using a horizontal, water-cooled bandsaw. A quarter inch slice was then taken approximately two inches from the ends of the two-inch-high step. The pieces were then further cut using a wet abrasive saw until the desired location was harvested in a mountable size (Figure 2.10) .

Figure 2.10 Micrograph locations on step bar taken from the center of the stepbar(polished surface is in the plane of the paper).

2.5.3 Y-Block Mold

The bottom inch of each y-block was cut-off and used to turn one tensile bar. The metallography samples from the y-blocks were removed from the grip of the tensile bars after they were pulled. The section removed for the tensile bar is shown by the dashed line in Figure 2.11.

Figure 2.11 Y-block showing the 1 inch section removed to be machined into a tensile bar

2.6 Metallography

Samples were taken from the tensile bar casting, y-block, and step bar as shown in Figures 2.9, Figure 2.10, and from the grip of the y-block tensile bar, respectively. These samples were mounted in epoxy and polished on a Leco AP-300 9 sample autopolisher per the polishing procedure in Table 2.1.

Table 2.1 Metallographic Preparation Procedure

Grit	RPM	Pressure (psi)	Time (sec)
120	300	50	Until Flat
180	300	50	120
300	300	50	120
600	250	40	120
800	260	30	120
1200	240	25	120
1-micron diamond	150	15	150

2.6.1 Graphite Analysis

Nodularity was measured using image analysis with ImageJ (Rasband). A macro was written for this program by a former graduate student that determines the roundness of a particle, counts the number of particles above 50% round, and uses this information to determine the nodule density and the nodularity. Each sample had 5 unetched micrographs analyzed to quantify the average graphite morphology. The photos were taken with the microscope eye piece at 10X magnification and the objective at 5X resulting for a total of 50X magnification looking through the microscope. For the graphite analysis, the image is taken with the microscope camera with a magnification is 108X with the 5X objective.

2.6.2 Pearlite Content

The pearlite content was determined after etching each sample for thirty seconds with 2% nital. The etched samples were also photographed at 108X magnification. The pearlite content was determined using point counting with a 9x11 grid. Using this grid, an intersection on pearlite was counted as 1, and if it was on the border of the pearlite microconstituent it was counted as 0.5. The total number of intersections on pearlite was divided by 99 points to get an estimate of how much of the area was covered by pearlite. This was done with 10 fields of view for each sample.

2.7 Electron Microscopy

Electron microscopy was performed using a Philips XL 40 ESEM. The same samples used for optical microstructural analysis were also used for scanning electron microscopy. Images of the samples were collected using secondary electron imaging for topography and backscatter electron analysis was used to view compositional changes. A thin-window energy dispersive spectrometer (EDS) was used to gather qualitative composition data.

2.8 Mechanical Testing

The tensile bar samples were from the areas indicated in 2.4. For Alloy Set A, 6 tensile bars from the cylinder mold were tested per heat. For Alloy Set B, 6 tensile bars from the cylinder molds and 3 tensile bars from the y-blocks were tested. For Alloy B-Cu30-Mn40-L, only the 3 y-block bars were tested. These bars adhere to the ASTM B557 (Figure 2.11). These bars were pulled at a strain rate of 10^{-3} s^{-1} . A two-inch extensometer was used and with a 200,000 lb force load cell.

Figure 2.12 Tensile bar schematic (in inches).

3 Results

3.1 Chemical Analysis

The chemical analysis for Alloy Set A is shown in Table 3.1. The Cu and Mn values were typically on target, but the silicon values varied. The targeted copper and manganese values are listed in the alloy name, if the alloy was intending to have 0.15% Cu and 0.30% Mn it would be named A-Cu15-Mn30. A more detailed table is in Appendix B.

Table 3.1 Chemical Composition of Alloy Set A

Alloy	C	OES Si	Calculated Mold Si	CE	Cu	Mn
A-Cu05-Mn15	3.44	3.90	4.00	4.74	0.05	0.15
A-Cu05-Mn30	3.14	4.03	4.13	4.48	0.05	0.28
A-Cu05-Mn40	3.17	4.10	4.20	4.54	0.05	0.37
A-Cu10-Mn30	3.20	4.12	4.21	4.57	0.10	0.28
A-Cu15-Mn15	3.31	3.99	4.10	4.64	0.14	0.17
A-Cu08-Mn30	3.09	3.80	3.9	4.36	0.08	0.27
A-Cu10-Mn40	3.17	4.22	4.30	4.58	0.11	0.38
A-Cu15-Mn20	3.20	4.16	4.24	4.59	0.15	0.19
A-Cu15-Mn30	3.13	4.07	4.17	4.49	0.15	0.27
A-Cu20-Mn20	3.15	4.27	4.35	4.57	0.22	0.20

The chemical analysis for Alloy Set B is shown in Table 3.2. The silicon and carbon chemistries were closer to the targeted values. The naming convention for these alloys follows the same scheme as Alloy Set A. The exception is the B-Cu30-Mn40 alloy which was cast twice, once with high silicon (H), and once with low silicon (L). The carbon values with an asterisk were determined by thermal analysis and not combustion.

Table 3.2 Chemical Composition for Alloy Set B

Alloy	C	Si	CE	Cu	Mn
B-Cu05-Mn15	3.15	4.27	4.57	0.06	0.16
B-Cu30-Mn15	3.20	4.23	4.61	0.32	0.14
B-Cu05-Mn40	3.18	4.01	4.52	0.07	0.38
B-Cu18-Mn28	3.18*	4.04	4.53	0.19	0.27
B-Cu30-Mn40-H	3.10*	4.06	4.53	0.31	0.38
B-Cu30-Mn40-L	3.21*	3.83	4.47	0.31	0.37

3.2 Graphite Morphology

For Alloy Set A the tensile bar blank location (“T”) was examined for graphite morphology (Table 3.3). For Alloy Set B, the graphite morphology was assessed in 4 different locations (**Error! Reference source not found.** through Table 3.5): sections from the tensile bar mold “T”, the quarter inch step of the step bar “A”, the last to solidify region of the step bar “D”, and the grip of the y-block tensile bar “Y”. A representative unetched micrograph of each alloy is in Appendix C.

Table 3.3 Alloy Set a Graphite Morphology at Location "T"

Alloy	Nodularity	Nodule Density (mm ⁻¹)	Graphite %
Cu05-Mn15	82	129	3
Cu05-Mn30	80	181	4
Cu05-Mn40	88	234	5
Cu10-Mn30	77	115	2
Cu15-Mn15	87	330	7
Cu08-Mn30	66	148	4
Cu10-Mn40	78	99	2
Cu15-Mn20	89	81	3
Cu15-Mn30	71	152	4
Cu20-Mn20	85	277	6

Table 3.4 Graphite Nodularity for various locations from Alloy Set B

Location	A	D	T	Y
B-Cu05-Mn15	91	94.2	86	87.8
B-Cu30-Mn15	91.4	93.4	87.8	94.2
B-Cu05-Mn40	89.8	91.6	88.6	91.8
B-Cu18-Mn28	91.2	90.4	90.2	90
B- Cu30-Mn40-H	88.4	93	89.2	86.8
B- Cu30-Mn40-L	91.2	92.8	90.2	92.2

Table 3.5 Graphite nodule density (nodules per mm²) for various locations from Alloy Set B

Location	A	D	T	Y
B-Cu05-Mn15	283.8	291.8	323.6	312
B-Cu30-Mn15	235.6	269.8	269.4	270.6
B-Cu05-Mn40	224.2	246	348.4	301
B-Cu18-Mn28	296.4	269.4	298.8	276.4
B- Cu30-Mn40-H	258.6	273.4	336.6	340.8
B- Cu30-Mn40-L	311.2	251.2	303	452.2

Table 3.6 Graphite area percent for various locations from Alloy Set B

Location	A	D	T	Y
B-Cu05-Mn15	8.4	8.2	10.6	8.8
B-Cu30-Mn15	7.8	8.2	6.8	8.4
B-Cu05-Mn40	6.8	8.8	9.4	9.6
B-Cu18-Mn28	8.4	12	8.6	8.6
B- Cu30-Mn40-H	7.6	8.4	10.6	9.0
B- Cu30-Mn40-L	8.6	7.6	8.6	8.6

3.3 Matrix Microstructure

All the samples were primarily ferritic, but some alloys had small regions of pearlite. For the average pearlite by area for Alloy Set A (Table 3.7) only alloys A-Cu08-Mn30 and A-Cu05-Mn15 had any measurable pearlite.

Table 3.7 Pearlite content of the matrix for Alloy Set A

Alloy	Pearlite %
Cu05-Mn15	0 . 2
Cu05-Mn30	0
Cu05-Mn40	0
Cu10-Mn30	0
Cu15-Mn15	0
Cu08-Mn30	0 . 4
Cu10-Mn40	0
Cu15-Mn20	0
Cu15-Mn30	0
Cu20-Mn20	0

In Alloy Set B most of the samples had pearlite present (Table 3.8). All samples with 0% pearlite had no pearlite visible except for Sample B-Cu30-Mn15 which had one very small pearlite colony that was too small to be counted by point counting.

Table 3.8 Pearlite content by location for Alloy Set B (* indicates pearlite present but near 0%)

Location	A	D	T	Y
B-Cu05-Mn15	0.0	0.0	0.4	0.0
B-Cu30-Mn15	0*	0.7	2.1	0.0
B-Cu05-Mn40	0.1	0.3	2.4	0.0
B-Cu18-Mn28	0.2	2.1	1.3	0.1
B- Cu30-Mn40-H	0.2	3.4	3.3	0.5
B- Cu30-Mn40-L	3.0	7.0	8.4	0.4

3.4 Mechanical Testing

Six tensile bars from the tensile bar molds were pulled for each alloy. The targeted grade is GJS-500-14 (Table 1.1), so the ultimate tensile strength (UTS) should meet or exceed 500 MPa, the yield should meet or exceed 400 MPa, and the elongation should meet or exceed 14%. As the standard for this iron is a European standard, the data is presented in SI units (Table 3.9).

Table 3.9 Tensile bar properties for Alloy Set A from the tensile bar blank casting

Name	UTS (MPa)	Yield (MPa)	% elong
Cu05-Mn15	550	442	10.0%
Cu05-Mn30	595	489	11.5%
Cu05-Mn40	592	500	9.4%
Cu10-Mn30	602	501	11.1%
Cu15-Mn15	602	500	11.4%
Cu08-Mn30	558	441	10.1%
Cu10-Mn40	616	517	8.4%
Cu15-Mn20	603	501	8.9%
Cu15-Mn30	596	491	11.9%
Cu20-Mn20	627	528	8.3%

In Alloy Set B, 6 tensile bar from each tensile bar mold were also tested (Table 3.10). Alloy B-Cu30-Mn40-L was not tested using cast rod tensile bars.

Table 3.10 Tensile properties of Alloy Set B from the tensile bar blank casting

Sample	Peak Stress	Stress At Offset Yield	Strain At Break
B-Cu05-Mn15	594	489	11.4%
B-Cu30-Mn15	596	489	8.4%
B-Cu05-Mn40	595	476	12.3%
B-Cu18-Mn28	584	471	12.3%
B-Cu30-Mn40-H	599	482	9.2%

Alloy Set B was also cast into y-block mold. The bottom inch was used to made tensile bars. The y-blocks had much higher elongations compared to the tensile bars from the tensile bar molds (Table 3.11).

Table 3.11 Tensile properties for Alloy Set B using samples cut from the bottom of the y-block castings

	UTS (MPa)	Yield (MPa)	% elong
B-Cu05-Mn15	571	465	18.4%
B-Cu30-Mn15	574	474	20.3%
B-Cu05-Mn40	553	448	19.4%
B-Cu18-Mn28	553	447	19.5%
B-Cu30-Mn40-H	554	448	20.0%
B-Cu30-Mn40-L	538	427	20.8%

4 Discussion

4.1 Alloy Set A

4.1.1 Microstructure

The Alloy Set A microstructure had virtually no pearlite even at the higher residual levels of Mn and Cu. The two samples, A-Cu05-Mn15 and A-Cu08-Mn30, that had pearlite were both under 4% silicon. Alloy A-Cu05-Mn15 had 0.2% pearlite with low residuals while higher residual alloys, such as A-Cu10-Mn30 had no pearlite. No alloys in Alloy Set A exceeded the 5% maximum of pearlite allowed per the DIN standard [3]

4.1.2 Tensile Properties

Alloy Set A tensile bars were taken from the rod castings. This method was preferred because each mold yielded 4 rods and which were faster to turn into tensile bars, as the round shape did not require rough machining. Based on the tensile properties for UTS and yield the iron easily met the specified GJS-500-14 grade, exceeding 500 MPa and 400 MPa respectively. Visually it is difficult to determine a correlation between silicon (Figure 4.1) manganese (Figure 4.2), or copper (Figure 4.3) for the yield or the UTS. A regression analysis in Minitab 18 fit equations to the yield (2) and UTS (3) as a function of chemistry. The R squared value for the yield equation was 98% and the UTS was 83%.

$$\begin{aligned} \text{Yield (MPa)} = & 2514 - 591.7(\text{Si}) - 9505(\text{Mn}) + 1686(\text{Cu}) - 3243(\text{Si})^2 + \\ & 2905(\text{Si})(\text{Mn}) - 6322(\text{Mn})(\text{Cu}) \end{aligned} \quad (2)$$

$$\begin{aligned} \text{UTS (MPa)} = & -183 + 179.0(\text{Si}) + 131.0(\text{Mn}) + 3244(\text{Cu}) - 691(\text{Si})(\text{Cu}) - \\ & 1082(\text{Cu})(\text{Mn}) \end{aligned} \quad (3)$$

Figure 4.1 Yield and UTS vs silicon content of Alloy Set A.

Figure 4.2 Yield and UTS vs copper content of Alloy Set A.

Figure 4.3 Yield and UTS vs the manganese content of Alloy Set A.

The elongations were not consistently high enough to meet the 14% elongation required for the alloy. There was large variation in the elongation of the bars (Figure 4.4). Some of the tensile bar fractures showed evidence of slag inclusions. No relationship between composition and elongation was observed for Alloy Set A.

Figure 4.4 Elongation at failure vs silicon content for Alloy Set A.

The yield to UTS ratio for ranged from 0.79-0.84 (Figure 4.5), . The average yield to UTS ratio was 0.83, much higher than the reported 0.55 for traditional ductile iron, as the higher silicon content increased the yield to UTS ratio [12]. The regression analysis for this data was only able to account for 61% of the variation, with silicon being the largest factor.

Figure 4.5 Yield to UTS ratio vs silicon content for Alloy Set A.

4.1.3 Alloy Set A Lessons Learned

Alloy Set B was designed based off of the information learned from Alloy Set A. Alloy Set A did not exceed the 5% pearlite maximum stated in the standard and all the samples contained very little pearlite. In an effort to produce more pearlite in the iron the amounts of alloying additions added to the iron was increased. An attempt was also made to better control the amount of silicon in the iron though updating the thermal analysis program and employing an onsite optical emission spectrometer measure silicon during the process.

The tensile properties produced by Alloy Set A, particularly the elongation, were less than expected based off of literature (Bjorkgren 2000) for standard grades of SSFDI irons. Poor elongation results were attributed to inclusion in the tensile round casting and filters were added for the Alloy Set B pours. Y-blocks were also added to the pour to insure proper metal feeding, a slower cooling rate, and tensile bars free from slag inclusions.

4.2 Alloy Set B

4.2.1 Microstructure

4.2.1.1 Optical Metallography

Alloy Set B castings contained more pearlite than those of Alloy Set A. The T samples from the tensile bar rounds and D samples from the center of the step bar had the highest amounts of pearlite. This was unexpected because they have lower cooling rates than Alloy Set A which had little pearlite. In traditional grades of ductile iron faster cooling rates promote pearlite which is a main contributor to the inhomogeneous mechanical properties in traditional ductile iron. Thinner sections typically contain more pearlite and have a higher hardness [8].

The pearlite content of the alloys was analyzed with multiple regression using the factors of silicon, copper, and manganese. A separate equation was generated for each micrograph sample location. Based on the regression model the manganese level was not a significant contributor to the pearlite in any of the sample areas. The equations generated for each location are self-consistent, with the main difference being the strength of the copper effect on pearlite promotion. The R-squared values for this analysis was 93.18 location “A” corresponds with equation 4, likewise “D” is 5, location “T” is 6, and location “Y” is equation 7.

$$\%P = 468 - 225.0(Si) + 1.46(Cu) + 27.04(Si)^2 \quad (4)$$

$$\%P = 493 - 231.1(Si) + 9.03(Cu) + 27.04(Si)^2 \quad (5)$$

$$\%P = 500 - 232.2(Si) + 7.97(Cu) + 27.04(Si)^2 \quad (6)$$

$$\%P = 448 - 220.2(Si) + 0.18(Cu) + 27.04(Si)^2 \quad (7)$$

Figure 4.6 Cooling rate ($^{\circ}\text{C/s}$) for cylinder mold; micrographs from the $4\text{ }^{\circ}\text{C/s}$ section, location “T”.

Figure 4.7 Cooling rate ($^{\circ}\text{C/s}$) of largest step in step bar casting; the center is the last to freeze section cooling at 1.1°C/s , location “C”.

Figure 4.8 Cooling rate ($^{\circ}\text{C/s}$) of quarter-inch step of step bar casting; this is one of the first to freeze regions with a cooling rate of 11°C/s , location “A”.

MAGMA

Figure 4.9 Cooling rate ($^{\circ}\text{C/s}$) of the Y-block casting; this is one of the first to freeze regions with a cooling rate of 1.2°C/s , location “Y”.

Although A and Y have different cooling rates, these locations are near the first to freeze in their respective castings. Location D in the step bar is the last to freeze section, while location T in the tensile bar rounds is near the last to freeze section of the casting. Based on the effect of copper on pearlite being largest in the last to freeze sample of the casting and it being smallest in the first to freeze section of the casting it is likely that there is copper segregation occurring as the metal is solidifying.

Table 4.1 Cooling rate and freezing sequence of micrograph locations

Micrograph Location	Casting	Cooling Rate (°C/s)	Freezing Sequence
A	Step Bar	11	Near First
D	Step Bar	1.1	Last
T	Tensile Rod	4	Near Last
Y	y-block	1.2	Near First

An ANOVA General Linear Model using the factors shown in Table 4.2 with pearlite content as the response, in which insignificant terms were excluded iteratively. The inputs were rounded to nominal factor levels. As the 4 micrographic samples came from 3 different castings, the freezing order was approximated as either 1) near the first to freeze for each respective castings or 2) near the last to freeze.

Table 4.2 Factors for the ANOVA GLM

C (wt%)	Si (wt%)	Cu (wt%)	Mn (wt%)	Cooling Rate (°C/s)	Freezing Order
3.20	4.25	0.05	0.15	1.13	1
3.15	4.0	0.185	0.275	1.5	2
3.10	3.8	0.30	0.40	2.4	
				10.0	

Silicon content and freezing order were identified as the most significant factors, as well as the interaction between silicon and the freezing order. The statistical analysis for the run is presented in Appendix D. Both silicon and the freezing order had a p-value of 0 while the interaction term had a p-value of 0.001, all of which were statistically significant. The R-squared term adjusted for this number of data points is 84%, which is a good correlation. Higher silicon levels and first to freeze regions had lower pearlite volume fractions (Figure 4.10). A key observation in the interaction plot is that at later freezing times and low silicon levels the pearlite fraction is higher (Figure 4.11).

Figure 4.10 Main effects plot of silicon and the freezing order on pearlite content.

Figure 4.11 interaction plot illustrating that as the silicon decrease the amount of pearlite increases and that it increased the pearlite less in a first to freeze section.

The increased pearlite in last to freeze or areas with a lower cooling rate is also seen in research by Moritz Riebisch from the RWTH Aachen University in Aachen Germany [15,16]. In these studies, SSFDI heats were alloyed with carbide promoting elements found in carbidic steels and with copper. The alloys in this work show a decreased pearlite percentage compared to that in the Riebisch with similar manganese and copper contents.

Table 4.3 Results from the Experiments of Riebisch

Alloy	C	Si	CE	Cu	Mn	Nodularity	Nodule Density (mm⁻¹)	Pearlite %
Cu 1.1	2.95	4.05	4.30	0.061	0.109	77.2	656	2.8
Cu 1.2	2.78	3.84	4.06	0.179	0.105	82.3	504	5.4
Cu 1.3	3.12	3.91	4.42	0.111	0.227	81.0	617	4.3
Cu 1.4	2.77	3.94	4.08	0.114	0.269	84.5	457	13.6
Cu 2.1	3.32	4.04	4.67	0.062	0.169	87.2	526	3.2
Cu 2.2	3.24	4.15	4.62	0.094	0.161	91.9	489	5.0
Cu 2.3	3.26	4.22	4.67	0.281	0.181	83.0	437	0.7
Cu 2.4	3.17	4.14	4.55	0.300	0.163	90.7	426	15.6
Mn 1	3.02	4.02	4.36	-	.172	76	415	2.0
Mn 2	2.95	3.82	4.22	-	.519	88	421	6.0

4.2.1.2 Electron Microscopy

To further investigate why the step bars specimens show an increased pearlite content in the last to cool sections of the casting EDS was performed using an environmental SEM equipped with EDS. It was not possible to discern pearlite or any compositional variation in the unetched samples. With the etched samples the pearlite could be seen topographically using secondary electron imaging (Figure 4.12). The pearlite is also visible in the backscatter imaging but the color variation shown in this image is more likely due to tunneling effects and the grain orientation than compositional differences (Figure 4.13).

Figure 4.12 Secondary electron image of pearlite colony in sample B-Cu30-Mn40-L at location "C", etched with nital.

Figure 4.13 Backscatter electron image of pearlite colony in sample B-Cu30-Mn40-L at location "C", etched with 2% nital.

Element mapping was performed on the area containing pearlite in the etched sample and the resulting image is shown in (Figure 4.14). It is just possible to discern that in the area where the pearlite is present there is a drop in silicon (green) and an increase in manganese (red), the copper (blue) seems to be evenly dispersed throughout the whole image. The bright green lines contain silicon and bismuth (determined by spectra from a point analysis) and were caused by the inoculant used in this iron.

Figure 4.14 EDS element map of pearlite colony in sample B-Cu30-Mn40-L at location "C", etched with 2% nital, Si is green, Mn is red, and Cu is blue.

It is unclear if this segregation of the silicon and manganese is what is causing the increase in pearlite content in the last to solidify areas of the casting, because the same effect is seen in the "A" section of the step bar which is nearly the first to freeze material (Figure 4.15, Figure 4.16, Figure 4.17).

Figure 4.15 Secondary electron image of pearlite colony in sample B-Cu30-Mn40-L at location "A", etched with nital.

100 μ m

Figure 4.16 Backscatter election image of pearlite colony in sample B-Cu30-Mn40-L at location "A", etched with 2% nital.

Figure 4.17 EDS element map of pearlite colony in sample B-Cu30-Mn40-L at location "A", etched with 2% nital, Si is green, Mn is red, and Cu is blue.

4.2.1.3 Scheil Analysis

The Scheil method provides a thermodynamic model of how the liquid composition is altered as some elements form the solid phases. This method uses the assumptions that the liquid is perfectly mixed at all times and that there is no diffusion in the solid phase. The computing of the composition of the liquid for all of the alloys was generated in Thermo-Calc 2018b and the TCFE9 database, the compositional inputs were the carbon, silicon, manganese, and copper values determined by the OES chemical analysis for each alloy with the balance of the materials being iron.

A graph showing the composition of silicon in the liquid for each of the Alloy Set B chemistries of iron is shown in Figure 4.18. The graph shows the silicon content in the liquid iron between the liquidus and solidus temperatures, the line starts at the liquidus temperature and ends at the solidus. As the metal cools down (right to left) the silicon in the liquid is depleted. Franzen discusses in his paper *Modification of Silicon Microsegregation in Solid Solution Strengthened Ductile Iron by Alloying with Aluminum* that the silicon has the highest concentration right around the graphite nodules, and that this concentration can exceed the 4.3% value at which point embrittlement due to B2-superstructures can occur [17]. If the silicon is congregated around the graphite nodules

then there is less in the remaining melt, if there is less silicon in this last to freeze iron, there will be less suppression of carbides. Because the manganese content in the melt remains relatively constant at these temperatures while the silicon decreases there is not enough silicon to continue to suppress formation of pearlite. This connects well with the earlier DOE work that showed silicon and freezing order were the main factors and that if there is higher silicon the order of cooling matters less.

Figure 4.18 Silicon content in the liquid iron as the casting cools according to the Scheil method.

All of the alloys retain most of the manganese in the liquid until the eutectic transformation, when all of the remaining liquid transforms to a solid. Alloys B-Cu30-Mn40-H, B-Cu30-Mn40-L, and B-Cu18-Mn28 have a very sharp drop in silicon as they approach the solidus temperature. This drop appears to start around 1175 °C in the alloys with Cu30-Mn40 and it occurs around 1173 °C in the alloys in B-Cu18-Mn28 and B-Cu05-Mn15. This drop starts in B-Cu30-Mn15 earlier around 1176 °C. Alloy B-Cu05-Mn40 had the highest solidus temperature and no drop was calculated.

4.2.2 Mechanical Properties

4.2.2.1 Y-Blocks

Six rod castings were again machined into tensile bars, and in addition 3 y-blocks were made into tensile bars. Differences between the two sample types were significant based

on a one-way ANOVA analysis. For UTS, yield, and elongation the y-blocks produced statistically different tensile properties with all of the p-values for these comparisons were equal to zero. The ratio of the yield to UTS was not statistically different from the tensile round casting to the y-block casting, this had a p-value of 0.898.

The y-block tensile bars had statistically less variance than that of the tensile round bars for elongation, using a test of equal variance the p-values was 0, as the p-value is less than 0.05, the null hypothesis (that they are the same) is rejected (Figure 4.19). The yield strength had a statistically different variance that was smaller in the tensile round bars. The UTS and yield/UTS variance were not statistically different. Due to the differences in the tensile properties between the two sample types and because the y-block sample was based on the ASTM standard and produced better mechanical properties, the y-block samples were used in the analysis. Alloy B-Cu30-Mn40-L was produced after the findings on the effectiveness of the tensile bar round mold and only had y-block samples machined and tested for tensile properties.

Figure 4.19. Test for equal variances for elongation between the y-block tensile bar and the tensile bars from the rod mold.

Figure 4.20. Test for equal variances for yield between the y-block tensile bar and the tensile bars from the rod mold.

Figure 4.21. Elongation in the tensile rods vs elongation in the y-block

4.2.2.2 Tensile Testing

The y-block tensile data was analyzed using regression analysis using silicon, manganese, and copper content as factors. An equation for UTS (8) was generated and the only significant factor was silicon; R-squared was 81% which means that 81 percent of the observed variation is accounted for by silicon content. The yield strength model (9) used all three elements, and R-squared was 87% (Figure 2.10). The elongation regression had a R-squared value of 68% and the effects of silicon and copper were significant. The yield/UTS model (10) accounted for 83% of the variation and silicon and copper were significant factors.

$$UTS(MPa) = 223.6 + 81.91(Si) \quad (8)$$

$$Yield(MPa) = 38.0 + 97.4(Si) + 91.4(Cu) + 52.8(Mn) - 283(Cu)(Mn) \quad (9)$$

$$Yield/UTS = 0.7896 + 0.0050(Si) - 0.989(Cu) + 0.2445(Si)(Cu) \quad (10)$$

5 Conclusions

Two sets of SSFDI alloys were cast with intentionally varying copper and manganese values. These alloy sets were used to create tensile bars and photomicrographs to determine the effect of the copper and manganese on the pearlite content and mechanical properties of the SSFDI.

In these high silicon alloys more pearlite was formed in thick sections of the casting. This effects is related to the silicon content and the least to freeze order in the castings.

5.1 Hypothesis

The SSFDI iron was able to tolerate combined copper and manganese values of 0.3 and 0.4 respectively with a 3.8% silicon level while maintaining mechanical properties necessary to meet the 500-14 grade, the pearlite content, however, exceeded the maximum of 5% pearlite in some areas of the castings.

With a further increases in silicon content to 4.06% the same levels of copper and manganese were tolerated without exceeding the maximum allowable pearlite content of 5%. These levels of alloying additions in traditional grades of ductile iron would result in a mixed ferrite pearlite microstructure.

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7 Charge Composition Alloy Set B

Table 7.1 Charge Sheet for Alloy B-Cu05-Mn15

Alloy B-Cu05-Mn15			Project Heat 17							MTU Alloy D180927							
Targets	Comp	CE	C	Si	Mg	P	Mn	S	Al	Cu	Ni	Ti	Cr	Ca	RE	Bi	Ba
	Lower	4.401	3.15	3.75	0.03	0.015	0.13	0		0.03							
	Target	4.472	3.2	3.8	0.04	0.015	0.15	0.01		0.05							
	Upper	4.653	3.3	4	0.05	0.025	0.17	0.04		0.07	2						
	Material	Weight (lbs)	C	Si	Mg	P	Mn	S	Al	Cu	Ni	Ti	Cr	Ca	RE	Bi	Ba
Furnace Additions	Steel Punchings	15	0.09	0.08		0.008	0.396	0.013		0.024	0.013						
	Pig Iron - Waupaca	60.5	4.38	0.62		0.041	0.03	0.027		0.0005	0.003	0.006	0.004				
	Waupaca 9/17 Risers	55	3.7	2.5		0.031	0.29	0.004	0.022	0.12	0.022	0.006	0.174				
	D180920 pig	118.75	3.3	3.95		0.048	0.165			0.07							
	FeMn (HW)		6.38	0.47		0.185	76.28	0.01									
	Cu									100							
	Desulco 9001		99.7					0.03									
	Foundrisil - charge	2.5		73.98		0.013			0.6					0.2	0.006		1.06
	Furnace Weight	251.75	3.320	3.199	0	0.040	0.172	0.008	0.011	0.061	0.006	0.003	0.039	0.002	0.000	0.000	0.011
			CE =	4.398													
Ladle Additions	HW - 5LB MgFeSi	2		44.4	6.44				0.81					1.03	0.93		
	Spherix	1		72					0.9					1.5	0.5	1	
	Topseed	1.083	0.094	49.04					1.038					0.996			1.03
	Cover Steel	2.67	0.18	0.15		0.008	0.467	0.013		0.024	0.013						
	Treated Total	258.503	3.236	3.803	0.042	0.039	0.172	0.008	0.025	0.059	0.006	0.003	0.038	0.020	0.007	0.004	0.015
			CE =	4.515													

Table 7.2 Charge Sheet for Alloy B-Cu30-Mn15

Alloy B-Cu30-Mn40			Project Heat 16							MTU Alloy D180921							
Targets	Comp	CE	C	Si	Mg	P	Mn	S	Al	Cu	Ni	Ti	Cr	Ca	RE	Bi	Ba
	Lower	4.40	3.15	3.75	0.03	0.015	0.13	0		0.28							
	Target	4.47	3.2	3.8	0.04	0.015	0.15	0.01		0.3							
	Upper	4.65	3.3	4	0.05	0.025	0.17	0.04		0.32	2						
	Material	Weight (lbs)	C	Si	Mg	P	Mn	S	Al	Cu	Ni	Ti	Cr	Ca	RE	Bi	Ba
Furnace Additions	Steel Punchings	46	0.09	0.08		0.008	0.396	0.013		0.024	0.013						
	Pig Iron - Waupaca	161.5	4.38	0.62		0.041	0.03	0.027		0.0005	0.003	0.006	0.004				
	Waupaca 9/17 Risers	72.55	3.7	2.5		0.031	0.29	0.004	0.022	0.12	0.022	0.006	0.174				
	FeMn (HW)		6.38	0.47		0.185	76.28	0.01									
	Cu	0.82								100							
	Desulco 9001		99.7					0.03									
	Foundrisil - charge	8.75		73.98		0.013			0.6					0.2	0.006		1.06
	Furnace Weight	289.62	3.282	3.123	0	0.032	0.152	0.018	0.024	0.317	0.009	0.005	0.046	0.006	0.000	0.000	0.032
			CE =	4.337													
Ladle Additions	HW - 5LB MgFeSi	2.7		44.4	6.44				0.81					1.03	0.93		
	Spherix	1.2		72					0.9					1.5	0.5	1	
	Topseed	1.3	0.094	49.04					1.038					0.996			1.03
	Cover Steel	2.67	0.18	0.15		0.008	0.467	0.013		0.024	0.013						
	Treated Total	297.49	3.197	3.795	0.050	0.032	0.152	0.018	0.039	0.309	0.009	0.005	0.045	0.026	0.009	0.004	0.036
		Percent	CE =	4.476													

Table 7.3 Charge Sheet for Alloy B-Cu05-Mn40

Alloy B-Cu05-Mn40			Project Heat 18							MTU Alloy D180928							
Targets	Comp	CE	C	Si	Mg	P	Mn	S	Al	Cu	Ni	Ti	Cr	Ca	RE	Bi	Ba
	Lower	4.40	3.15	3.75	0.03	0.015	0.38	0		0.03							
	Target	4.47	3.2	3.8	0.04	0.015	0.40	0.01		0.05							
	Upper	4.65	3.3	4	0.05	0.025	0.42	0.04		0.07	2						
	Material	Weight (lbs)	C	Si	Mg	P	Mn	S	Al	Cu	Ni	Ti	Cr	Ca	RE	Bi	Ba
Furnace Additions	Steel Punchings	12	0.09	0.08		0.008	0.396	0.013		0.024	0.013						
	Pig Iron - Waupaca	39.3	4.38	0.62		0.041	0.03	0.027		0.0005	0.003	0.006	0.004				
	Waupaca 9/17 Risers	74.8	3.7	2.5		0.031	0.29	0.004	0.022	0.12	0.022	0.006	0.174				
	D180920 pig	70.65	3.3	3.95		0.048	0.165			0.07							
	D180927Pig	56.15	3.113	3.96			0.16			0.06							
	FeMn (HW)	0.75	6.38	0.47		0.185	76.28	0.01									
	Cu									100							
	Desulco 9001		99.7					0.03									
	Foundrisil - charge	1		73.98		0.013			0.6					0.2	0.006		1.06
	Foundrisil - top	1		74.9					1.04					1.04			1.06
	Furnace Weight	255.65	3.273	3.210	0	0.030	0.413	0.006	0.013	0.069	0.008	0.003	0.052	0.005	0	0	0.008
			CE =	4.344													
Ladle Additions	HW - 5LB MgFeSi	2		44.4	6.44				0.81					1.03	0.93		
	Spherix	1		72					0.9					1.5	0.5	1	
	Topseed	1.083	0.094	49.04					1.038					0.996			1.03
	Cover Steel	2.67	0.18	0.15		0.008	0.467	0.013		0.024	0.013						
	Treated Total	262.403	3.191	3.805	0.042	0.029	0.407	0.006	0.026	0.067	0.007	0.003	0.050	0.022	0.007	0.004	0.012
			CE =	4.461													

Table 7.4 Charge Sheet for Alloy B-Cu18-Mn28

Alloy B-Cu18-Mn28			Project Heat 20							MTU Alloy 181005							
Targets	Comp	CE	C	Si	Mg	P	Mn	S	Al	Cu	Ni	Ti	Cr	Ca	RE	Bi	Ba
	Lower	4.40	3.15	3.75	0.03	0.015	0.26	0		0.15							
	Target	4.47	3.2	3.8	0.04	0.015	0.275	0.01		0.175							
	Upper	4.65	3.3	4	0.05	0.025	0.3	0.04		0.2	2						
	Material	Weight (lbs)	C	Si	Mg	P	Mn	S	Al	Cu	Ni	Ti	Cr	Ca	RE	Bi	Ba
Furnace Additions	Steel Punchings	10	0.09	0.08		0.008	0.396	0.013		0.024	0.013						
	Pig Iron - Waupaca	63.85	4.38	0.62		0.041	0.03	0.027		0.0005	0.003	0.006	0.004				
	Waupaca 9/17 Risers	8.6	3.7	2.5		0.031	0.29	0.004	0.022	0.12	0.022	0.006	0.174				
	D180507 h13	34.05	3.2	4.16		0.048	0.19			0.15							
	D181004 H19	134.6	2.95	4.3			0.271			0.18							
	FeMn (HW)	0.24	6.38	0.47		0.185	76.28	0.01									
	Cu	0.15								100							
	Desulco 9001	0.35	99.7					0.03									
	Foundrisil - top	0.7		74.9					1.04					1.04			1.06
	Furnace Weight	252.54	3.283	3.207	0	0.018	0.276	0.008	0.004	0.181	0.002	0.002	0.007	0.003	0	0	0.003
			CE =	4.354													
Ladle Additions	HW - 5LB MgFeSi	2		44.4	6.44				0.81					1.03	0.93		
	Spherix	1		72					0.9					1.5	0.5	1	
	Topseed	1.083	0.094	49.04					1.038					0.996			1.03
	Cover Steel	2.67	0.18	0.15		0.008	0.467	0.013		0.024	0.013						
	Treated Total	259.293	3.200	3.810	0.042	0.018	0.273	0.007	0.018	0.176	0.002	0.002	0.007	0.021	0.007	0.004	0.007
			CE =	4.472													

Table 7.5 Charge Sheet for Alloy B-Cu30-Mn40-H

Alloy B-Cu30-Mn40-H			Project Heat 21							MTU Alloy D181009							
Targets	Comp	CE	C	Si	Mg	P	Mn	S	Al	Cu	Ni	Ti	Cr	Ca	RE	Bi	Ba
	Lower	4.39	3.15	3.75	0.03	0.015	0.38	0		0.28							
	Target	4.46	3.2	3.8	0.04	0.015	0.4	0.01		0.3							
	Upper	4.65	3.3	4	0.05	0.025	0.42	0.04		0.32	2						
	Material	Weight (lbs)	C	Si	Mg	P	Mn	S	Al	Cu	Ni	Ti	Cr	Ca	RE	Bi	Ba
Furnace Additions	Steel Punchings	10	0.09	0.08		0.008	0.396	0.013		0.024	0.013						
	Pig Iron - Waupaca	30	4.38	0.62		0.041	0.03	0.027		0.0005	0.003	0.006	0.004				
	Waupaca 9/17 Risers	60	3.7	2.5		0.031	0.29	0.004	0.022	0.12	0.022	0.006	0.174				
	D181005 H20	150.35	3.18	3.9		0.029	0.272			0.18							
	FeMn (HW)	0.55	6.38	0.47		0.185	76.28	0.01									
	Cu	0.47								100							
	Desulco 9001	0.2	99.7					0.03									
	Foundrisil - charge	0.5		73.98		0.013			0.6					0.2	0.006		1.06
	Furnace Weight	252.07	3.293	3.097	0	0.030	0.417	0.005	0.006	0.323	0.006	0.002	0.042	0.000	0.000	0	0.002
			CE =	4.326													
Ladle Additions	HW - 5LB MgFeSi	2		44.4	6.44				0.81					1.03	0.93		
	Spherix	1		72					0.9					1.5	0.5	1	
	Topseed	1.083	0.094	49.04					1.038					0.996			1.03
	Cover Steel	2.67	0.18	0.15		0.008	0.467	0.013		0.024	0.013						
	Treated Total	258.823	3.210	3.7031	0.0423	0.030	0.4109	0.0047	0.0203	0.3152	0.0061	0.0021	0.0408	0.0183	0.0072	0.0039	0.006
	Percent		CE =	4.445													

Table 7.6 Charge Sheet for Alloy B-Cu30-Mn40-L

Alloy B-Cu30-Mn40-L			Project Heat 22							MTU Alloy D181101							
Targets	Comp	CE	C	Si	Mg	P	Mn	S	Al	Cu	Ni	Ti	Cr	Ca	RE	Bi	Ba
	Lower	4.41	3.25	3.5	0.03	0.015	0.38	0		0.28							
	Target	4.50	3.3	3.6	0.04	0.015	0.4	0.01		0.3							
	Upper	4.60	3.35	3.7	0.05	0.025	0.42	0.04		0.32	2						
	Material	Weight (lbs)	C	Si	Mg	P	Mn	S	Al	Cu	Ni	Ti	Cr	Ca	RE	Bi	Ba
Furnace Additions	Steel Punchings	10	0.09	0.08		0.008	0.396	0.013		0.024	0.013						
	Pig Iron - Waupaca	22.9	4.38	0.62		0.041	0.03	0.027		0.0005	0.003	0.006	0.004				
	Waupaca 9/17 Risers	126.5	3.7	2.5		0.031	0.29	0.004	0.022	0.12	0.022	0.006	0.174				
	D181009 pig	94.55	3.1	4.062		0.036	0.378	0.016	0.018	0.312	0.021	0.01	0.021				
	FeMn (HW)	0.33	6.38	0.47		0.185	76.28	0.01									
	Cu	0.36								100							
	Desulco 9001	0.3	99.7					0.03									
	Foundrisil - charge	0.5		73.98		0.013			0.6					0.2	0.006		1.06
	Furnace Weight	255.44	3.396	2.857	0	0.033	0.400	0.011	0.019	0.317	0.019	0.007	0.094	0.000	0.000	0.000	0.002
			CE =	4.353													
Ladle Additions	HW - 5LB MgFeSi	2		45.66	6.06				0.93					0.98	1		
	Spherix	1		72					0.9					1.5	0.5	1	
	Topseed	1.083	0.094	49.04					1.038					0.996			1.03
	Cover Steel	2.67	0.09	0.08		0.008	0.4	0.013		0.024	0.013						
	Treated Total	262.193	3.310	3.470	0.039	0.032	0.394	0.011	0.033	0.309	0.019	0.007	0.092	0.018	0.008	0.004	0.006
	Percent		CE =	4.471													

8 Alloy Detailed Composition

Table 8.1 Combined Combustion Carbon and Sulfur Data with OES Chemistry

Alloy	C	Si	S	Mn	P	Mg	Ce	Cu	Cr	Mo	Sn	Ni	V	Ti	Pb	Al
B-Cu05-Mn15	3.15	4.22	0.013	0.16	0.036	0.046	0.0045	0.06	0.02	0.002	0.004	0.02	0.004	0.011	0.0005	0.013
B-Cu30-Mn15	3.2	4.26	0.018	0.14	0.036	0.054	0.0054	0.32	0.02	0.002	0.004	0.02	0.004	0.011	0.0012	0.016
B-Cu05-Mn40	3.18	3.99	0.01	0.39	0.033	0.042	0.0045	0.07	0.03	0.004	0.004	0.02	0.006	0.01	0.001	0.014
B-Cu18-Mn28	3.22	3.97	0.013	0.26	0.036	0.049	0.003	0.18	0.02	0.002	0.005	0.01	0.004	0.01	0.0005	0.013
B- Cu30-Mn40-H	3.17	3.96	0.009	0.38	0.03	0.045	0.0017	0.32	0.02	0.003	0.004	0.02	0.005	0.009	0.0008	0.013
B- Cu30-Mn40-L	3.26	3.77	0.009	0.36	0.03	0.053	0.0044	0.32	0.03	0.004	0.004	0.02	0.007	0.01	0.0012	0.015

9 Micrographs

Figure 9.1 B-Cu05-Mn15 etched with 2% nital from "A"

Figure 9.2 B-Cu05-Mn15 etched with 2% nital from "C"

Figure 9.3 B-Cu05-Mn15 etched with 2% nital from "T"

Figure 9.4 B-Cu05-Mn15 etched with 2% nital from "Y"

Figure 9.5 B-Cu30-Mn15 etched with 2% nital from "A"

Figure 9.6 B-Cu30-Mn15 etched with 2% nital from "C"

Figure 9.7 B-Cu30-Mn15 etched with 2% nital from "T"

Figure 9.8 B-Cu30-Mn15 etched with 2% nital from "Y"

Figure 9.9 B-Cu05-Mn40 etched with 2% nital from "A"

Figure 9.10 B-Cu05-Mn40 etched with 2% nital from "C"

Figure 9.11 B-Cu05-Mn40 etched with 2% nital from "T"

Figure 9.12 B-Cu05-Mn40 etched with 2% nital from "Y"

Figure 9.13 B-Cu18-Mn28 etched with 2% nital from "A"

Figure 9.14 B-Cu18-Mn28 etched with 2% nital from "C"

Figure 9.15 B-Cu18-Mn28 etched with 2% nital from "T"

Figure 9.16 B-Cu18-Mn28 etched with 2% nital from "Y"

Figure 9.17 B-Cu30-Mn40-H etched with 2% nital from "A"

Figure 9.18 B-Cu30-Mn40-H etched with 2% nital from "C"

Figure 9.19 B-Cu30-Mn40-H etched with 2% nital from "T"

Figure 9.20 B-Cu30-Mn40-H etched with 2% nital from "Y"

Figure 9.21 B-Cu30-Mn40-L etched with 2% nital from "A"

Figure 9.22 B-Cu30-Mn40-L etched with 2% nital from "C"

Figure 9.23 B-Cu30-Mn40-L etched with 2% nital from "T"

Figure 9.24 B-Cu30-Mn40-L etched with 2% nital from "Y"

10 General Linear Model

General Linear Model: Pearlite versus Si, Freezing Order

Method

Factor coding (-1, 0, +1)

Factor Information

Factor	Type	Levels	Values
Si	Fixed	3	3.80, 4.00, 4.25
Freezing Order	Fixed	2	1, 2

Analysis of Variance

Source	DF	Adj SS	Adj MS	F-Value	P-Value
Si	2	51.82	25.9124	32.22	0.000
Freezing Order	1	42.14	42.1399	52.40	0.000
Si*Freezing Order	2	18.87	9.4335	11.73	0.001
Error	18	14.48	0.8042		
Total	23	115.61			

Model Summary

S	R-sq	R-sq(adj)	R-sq(pred)
0.896792	87.48%	84.00%	71.20%

Coefficients

Term	Coef	SE Coef	T-Value	P-Value	VIF
Constant	2.077	0.202	10.26	0.000	
Si					
3.80	2.608	0.329	7.94	0.000	1.52
4.00	-0.923	0.252	-3.67	0.002	1.52
Freezing Order					
1	-1.465	0.202	-7.24	0.000	1.22
Si*Freezing Order					
3.80 1	-1.553	0.329	-4.73	0.000	1.61
4.00 1	0.479	0.252	1.90	0.073	1.57

Regression Equation

$$\begin{aligned}\text{Pearlite} = & 2.077 + 2.608 \text{ Si_3.80} - 0.923 \text{ Si_4.00} - 1.685 \text{ Si_4.25} - 1.465 \text{ Freezing Order_1} \\ & + 1.465 \text{ Freezing Order_2} - 1.553 \text{ Si*Freezing Order_3.80 1} \\ & + 1.553 \text{ Si*Freezing Order_3.80 2} + 0.479 \text{ Si*Freezing Order_4.00 1} \\ & - 0.479 \text{ Si*Freezing Order_4.00 2} + 1.074 \text{ Si*Freezing Order_4.25 1} \\ & - 1.074 \text{ Si*Freezing Order_4.25 2}\end{aligned}$$

Fits and Diagnostics for Unusual Observations

Obs	Pearlite	Fit	Resid	Std Resid	
10	0.303	2.140	-1.837	-2.24	R
21	2.980	1.667	1.313	2.07	R
24	0.354	1.667	-1.313	-2.07	R

R Large residual