Ductile Iron Quality Assurance Guide
by
Ductile Iron Society

Purposes of Quality Assurance Guide

The primary objective of this guide is to assist producers of ductile iron castings in obtaining higher levels of quality reliability.

It is not intended to be a complete textbook or handbook on ductile iron; these are available from several sources. (2) (3) (4) (20)

The primary intention is to:

- Guide management and supervision of new producers of ductile iron toward the controls, test, records and quality organization required to be a quality producer;
- Enlighten those considering ductile iron production to the controls and tests required;
- Guide long time producers toward higher levels of quality reliability and reduced variability.

Some explanations of metallurgy, processes, and variables will be given for better understanding of the necessary controls. More detailed explanations are included in Appendices in order to maintain the focus on control procedures in the main text.

Because of metallurgical sensitivity of ductile iron, major emphasis is focused on metal controls necessary for METALLURGICAL RELIABILITY. Since poor nodularity can cause more drastic loss of properties than in other metals, nodularity must be controlled. However, total casting quality requires sound castings free of shrinkage, porosity and dross, with good surface finish and dimensional accuracy. These are expected in other irons and metals, but since some of these defects are more complex in ductile iron, some brief guidance toward sound castings is included. Total casting quality is the ultimate objective.
CHAPTER 1

Quality Guidance – A Founding Purpose of the Ductile Iron Society

The Ductile Iron Society was organized for quality guidance. When the Inco patent was nearing expiration and license from the licensor was being phased out, need was recognized for some organization to provide quality guidance, training, technical meetings, and research to uphold metallurgical integrity, and casting quality, and to improve understanding and extend applications of ductile iron.

DIS published one of the first Quality Control Manuals, prepared by the Quality Assurance Committee over 25 years ago. This was an important early presentation of the metallurgical intricacies of ductile iron and controls necessary for its quality assurance.

Since that early manual, more processes have been developed, more precise knowledge gained, and quality expectations have been raised. This Guide is intended to update the first manual, retaining the original fundamentals, but including the advancements made and higher expectations of ductile iron quality.

To further encourage quality, a QUALITY CERTIFICATION PROGRAM was established by DIS over 25 years ago. Certification was an optional program in which the Technical Director made annual inspection visits to the foundry and audited the quality controls, test, records, and quality organization. If the met DIS standards the foundry was certified and given a Certified Producer name.

Following the initial guidance from DIS, most quality producers have developed their own Company Quality Control Manuals with details of their procedures, limits, tests, responsibilities, records, etc.

Manu purchasers of ductile iron castings have developed their own certification standards for auditing potential suppliers. The certification process has been extended to suppliers of materials to ductile iron producers.

RESEARCH PROJECTS OF THE DIS have been directed toward knowledge helpful toward better quality. A complete list of research projects can be found in Appendix I.

Briefly, research has been directed toward improving quality in the following areas:

- **BETTER STRUCTURAL CONTROL**
  - Factors influencing carbides
  - Influences on pearlite and nodularity
  - Optimum properties in heavy sections
  - Effect of trace elements on annealing time
  - Factors affecting nodule count

- **BETTER CASTING INTEGRITY**
  - Factors causing pin holes
  - Factors influencing dross
  - Factors affecting shrinkage

- **SPECIAL PROPERTIES**
  - Factors affecting machinability
Fracture toughness
Characterization of inclusions

Recommendations presented here toward better quality have come from:
- Information from research projects
- Experiences of quality producers on DIS Committees
- Consultant experiences, witnessing deficiencies and guiding improvements
- Purchaser auditing expectations

CHAPTER 2

Quality Controls More Critical On Ductile Iron (Executive Overview)

The meet structural expectations of ductile iron, more in-process controls, closer iron control, and more analyses and tests are necessary. These are briefly suggested below and explained in more detail under process steps.
- The outstanding properties of ductile iron are due to a nodular or spheroidal form of precipitated graphite in the microstructure.
- This favorable nodular graphite shape is obtained by treating with magnesium to retain residual Mg content within narrow control limits, then inoculating with ferrosilicon to nucleate that graphitization.
- Mg is a very volatile and reactive element and is difficult to control. Occasional “misses” are possible and likely.
- Many alloying and treatment methods have been used to successfully introduce Mg with various levels of recovery. But, all treatment processes are subject to iron variables, alloy variables, and mechanical variables which must be understood and controlled.
- The second step of ductile iron treatment is post inoculation with foundry grade FeSi to nucleate and encourage graphite precipitation. Good inoculation prevents formation of undesirable carbides which are very detrimental to ductility and also encourages good graphite form, size and distribution.
- Nucleation fades with time and castings should be poured within a time limit. Faded inoculation can be restored or boosted with a little inoculant in the stream or in the mold, using any of several addition techniques.
- Nodularity verification of each treatment batch is essential. The most positive verification is a quick microscopic examination of a specimen quickly polished and examined near the pouring floor. Ultrasonic instruments are also used to verify nodularity. Fig. 1 shows classification of graphite forms.
  Type I represents most spheroidal nodules. Type II nodules are less perfectly spherical but, in compact in form. Types III and IV are vermicular and crab like undesirable forms due to insufficient Mg or excessive tramp elements.
Type V is exploded graphite. A minimum of 80% nodular graphite shapes I and II in the casting is generally considered necessary to meet the expectations of ductile iron. With more than 20% vermicular, crab, or flake graphite, expected properties are not met. Batches with nodularity rating below 80% should be segregated before mixing in the shakeout and rejected.
(if retests are not acceptable). For safety parts, 90% minimum nodularity is usually required. Carbides can also be detected in the micro when etched. Any batches with carbides should be either rejected or reclaimed by heat treatment. Carbides in trace amounts can be very detrimental to ductility and machinability expected of ductile iron, and must be prevented.

- **Casting identification and traceability is essential first to separate acceptable and unacceptable nodularity and finally to match castings with all records of composition, and tests. Most foundries cast on, either a date, heat number, or code number that can be matched with records and tests.**

- **Since grades of ductile iron are based on combinations of tensile test properties, tensile bars are necessary to verify grade compliance.** For best control, tensile tests should be made on equipment in the foundry so results can be reported within one day, and decisions and corrections can be made. Some producers send test bars to a commercial laboratory for tensile tests. If results are not reported within a day or two, missed tests are sometimes not corrected, and adequate control is not maintained.

- **Through matrix control, several grades of ductile iron can be produced as cast, and more grades are possible by heat treatment. Ductile Iron provides a choice of several property combinations, a family of engineering materials.**

- **Controlling nodularity and matrix structure requires more select charge materials and closer control of melting and base composition.**

- **Metal analysis is essential to good control. A Spectrometer in the plant is almost essential for the chemical controls required.** Some new producers send samples to outside commercial laboratories that report analyses next day. Control is mode difficult when analyses cannot be reported promptly in the plant.

- Mg analysis is probably the second best control assurance. Acceptable nodularity usually requires Mg residual of 0.03% to 0.05% or 0.04% to 0.06%.

- Tramp element contamination can cause poor graphite within good Mg concentration. Monitoring of deleterious tramp elements is important.

- If Mg concentration is below minimum level, nodularity of graphite will drop below expected level. Unnecessarily high Mg contents increase tendency toward carbides, dross defects, and shrinkage.

- Closer control of base iron composition is essential for control of nodularity and matrix structure. For these reasons, spectrometer analyses are more important for in-process control of ductile iron, than on other irons and metals.

- Statistical Process Control (SPC) graphs and techniques are helpful toward the controls necessary for quality ductile iron. Highest quality producers utilize SPC techniques and most customer audits look for SPC programs as evidence of the quality controls they expect.

- Employee training courses are valuable toward developing understanding of the closer controls required. A sufficient number of trained personnel is important to insure quality reliability.

- Producers should develop their company Quality Control Manual which should contain the following:
  - Management commitment to quality.
  - Detailed procedures, controls, and limits on raw materials, melting, treatment, and casting, as well as sand control, inspection, and dimensional control.
  - Procedures for tests, micros, hardness, etc.
  - Clearly defined responsibility for key control personnel.
Adequate training of key control personnel.
- Responsibilities for gating, risering, sand control, dimensional checks, and final inspection.
- Standardization and calibration procedures for test equipment, gages, etc.
- Applications of SPC techniques for best control.
- Periodic audit of quality control procedures and provisions for revisions.

### Common Grades of Ductile Iron

More detailed listings of various specifications are included in Appendix II. ASTM specifications A 536 are most popular. For general understanding, the most common grades are listed in the following table:

#### Table 1. Common Grades of Ductile Iron

<table>
<thead>
<tr>
<th>Designation</th>
<th>BHN range</th>
<th>How produced</th>
<th>Matrix</th>
<th>Mn</th>
<th>Cu</th>
<th>Tin</th>
<th>Cr</th>
</tr>
</thead>
<tbody>
<tr>
<td>Ferritic High Ductility Grades</td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>60-40-18</td>
<td>149-187</td>
<td>Most anneal as cast</td>
<td>All ferrite</td>
<td>.20 max</td>
<td>.05 max</td>
<td>0</td>
<td>.05 max</td>
</tr>
<tr>
<td>65-45-12</td>
<td>156-217</td>
<td>Most as cast, few anneal</td>
<td>Ferrite 90%+</td>
<td>.35 max</td>
<td>.10</td>
<td>.01</td>
<td>.05</td>
</tr>
<tr>
<td>Pearlitic High Strength Grades</td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>80-55-06</td>
<td>187-255</td>
<td>As cast</td>
<td>Pearlite 40%-60%</td>
<td>.30-.60</td>
<td>.30-.70 or</td>
<td>.05-.08</td>
<td>.10 max</td>
</tr>
<tr>
<td>100-70-03</td>
<td>241-302</td>
<td>Normalized or more alloy</td>
<td>Pearlite 80%+</td>
<td></td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td></td>
<td></td>
<td></td>
<td>More of above alloys</td>
<td></td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>120-90-02</td>
<td>240-300</td>
<td>Quench &amp; Temper</td>
<td>Tempered martensite</td>
<td>Special</td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>Austempered Ductile Iron</td>
<td>UTS 125-230, E% 10-1, BHN 269-555</td>
<td>Specialized Heat Treatment</td>
<td>Specialized Heat Treatment</td>
<td></td>
<td></td>
<td></td>
<td></td>
</tr>
</tbody>
</table>

The matrix structures of the common grades of ductile iron consist of ferrite and pearlite, in various proportions. Ferrite, containing no combined carbon, has the lowest hardness, and highest ductility, with lower strength.

Pearlite, with combined carbon, contributes hardness and strength with lower ductility.

Fig. 2A shows the microstructure of a completely ferritic ductile iron with black graphite nodules in a white ferrite matrix with no pearlite. Fig. 2B shows a pearlitic grade with about 50% of gray pearlite that would meet 80-55-06 specification.

The high ductility grades require a maximum of ferrite and a minimum of pearlite. The highest ductility grade is 60-40-18 with:
- Minimum tensile strength - 60,000 psi
- Minimum yield strength - 40,000 psi
- Minimum elongation - 18%

Meeting 18% elongation requires a matrix of virtually all ferrite. Hardness range is roughly 149 to 187 BHN. Most producers anneal in order to more positively meet this specification. Some customers require annealing.
Fig 2A. Ferritic ductile iron

Fig 2B. Pearlitic ductile iron (as-cast)
Some foundries successfully produce this grade as-cast by concentrating on very select raw materials and by maintaining very low levels of manganese and alloy residuals. The 65-45-12 grade is a very popular and serviceable ferritic grade, which can have a little pearlite. Low concentrations of Mn and alloys are desirable but need not be as low as the higher ductility grade. Most producers meet these properties as-cast. Some producers anneal this grade rather than follow the closer metal controls required to meet as-cast.

The high strength grades require considerable pearlite which is encouraged by alloy additions of Cu or Sb. Since Mn encourages pearlite, higher Mn levels can be tolerated, as well as higher residuals of inadvertent trace alloys like Cr, V, Mo, etc. Less select charge materials are required for pearlitic high strength grades.

The 80-55-06 grade is a popular as-cast, high strength grade. Some additional alloying, with copper or tin, is usually required. Copper is a preferred alloy because it increases pearlite but does not increase carbide tendency. Tin also increases pearlite without increasing carbide tendency. Tin is very powerful, roughly 10 times as effective as copper. Because of its potency, tin requires closer controls and should be used only with closely calibrated Spectrometer monitoring. Otherwise, unrecognized tin residual can contaminated efforts to make ferritic ductile. Brinell hardness usually ranges 187-255 in this grade.

Many producers use a total alloy factor to determine how much additional copper (or tin) should be added.

The higher strength 100-70-03 grade can be met with about 80% or more pearlite. This pearlite level can be obtained with a higher total alloy combination. Or, the high pearlite level can be obtained with an air cool normalizing heat treatment. Hardness of this grade usually ranges 240-300 Brinell. A still higher strength grade 120-90-02 can be obtained by a quench and temper treatment, which produces a structure of tempered martensite. Alloys to enhance the heat treatment are helpful.

Returns from pearlitic and ferritic grades should be separated. Pearlitic returns in the charge for ferritic grades can introduce too much manganese and copper (or tin) to meet ferritic specifications as-cast, and require the cost of heat treatment. When pearlitic returns are separated and charged into a pearlitic heat the alloys can be utilized toward final alloy factor needed.

**AUSTEMPERED DUCTILE IRON (ADI)**

Austempered Ductile Iron is a super ductile iron with about twice the strength level and is obtained by specialized heat treatment. In highly specialized heat treatment equipment, castings are quenched into a molten salt bath, at a controlled temperature. By controlling temperature and time at austenitizing temperature and temperature of the salt bath, a range of super combinations of strength and ductility can be obtained with good impact and wear properties.

ASMT has established 5 grades of ADI ranging from 125,000 psi tensile strength with 10% elongation to 200,000 psi tensile with 1% elongation. A 230,000 tensile grade has no specified elongation.

With such attractive properties ADI castings have replaced steel forgings and castings in many engineering applications like gears, and high performance engine parts.
Good quality conventional ductile iron can be heat treated to ADI properties. On the other hand marginal quality ductile iron with either borderline nodularity, excessive inclusions, or microshrinkage, cannot be heat treated to the full expectations of ADI. Appendix V provides a more detailed explanation of Austempered Ductile Iron.

CHAPTER 3

CONTROLLING THE PROCESS STEPS – MELTING

After a brief overview or basic controls, tests, and quality expectations of ductile iron and common grades produced, this section will follow the various steps or production with more detailed explanations or variables encountered and controls needed. Melting is the first process requiring close control.

CLOSER MONITORING OF RAW MATERIALS

Every process starts with raw materials. Closer control of ray materials is required for quality ductile iron. All materials, including charge materials, recarburizers, treatment alloys, inoculants and all material affecting casting quality, should have specifications clearly agreed upon between the Purchasing Department and the Quality Control Manager or Metallurgist.

Situations have been encountered where the Purchasing Department of a company found a "good deal" in some charge material or a new source of treatment alloy which caused quality control problems.

Suppliers of materials and alloys should be encouraged to use Statistical Process Control techniques and provide reports of analysis and quality controls.

Specifications should be agreed upon within the foundry organization and thoroughly understood by the supplier.

Materials received should be inspected and analyses reviewed and filed where available for customer audit or company defense.

MORE SELECT CHARGE MATERIALS ARE REQUIRED in the production of ductile iron.

LOWER PHOSPHORUS CONTENT is essential. Below .05% phosphorus is needed and below .03% is desirable. Phosphorus lowers ductility which is the major attraction of ductile iron. Research is increasingly indicating better impact, fracture toughness, and fatigue strength with very low phosphorus contents.

LOW SULFUR CONTENT is desirable because magnesium reacts with sulfur which must be essentially removed before nodulizing begins. High sulfur content complicates magnesium control and generates sulfide inclusions which may not have time to float out.

Electric melting adds little or no sulfur and with good charge materials yields a sulfur level low enough for most treatment processes. The basic slag cupola can obtain sufficiently low sulfur contents.

The acid slag cupola requires any one of the several external desulfurizing processes to reduce sulfur to low enough concentrations before treatment.
LOW LEVELS OF TRACE ELEMENTS ARE IMPORTANT
Ductile Iron is sensitive to many trace elements even in low concentrations. These are described individually in Appendix VIII.

Table 2 gives a quick overview of the most serious contaminating tramp elements and low concentration at which trouble can be experienced.

The most serious are those SUBVERSIVE TO GRAPHITE NODULARITY like lead, antimony, and bismuth at low concentrations above 0.002%. Titanium which is beneficial toward good flake graphite structure in gray iron works against nodular graphite formation at 0.05% levels. Tellurium, zirconium, aluminum and zinc are also detrimental to nodularity.

Combinations of these elements can magnify their effects. However, cerium can neutralize somewhat and extend safe limits. There is much yet to be learned about combinations of these elements. The concentrations given are simply warnings that trouble may be experienced beyond these concentrations.

The next group of troublesome residuals can be called UNDESIRABLE CARBIDE FORMERS which are also pearlite formers. Boron is very powerful at the 0.002% level.

Chromium complicated ferritic grades at levels about 0.05%. Pearlitic grades may tolerate up to 0.10%.

Vanadium safe limits are 0.02% for ferritic and 0.05% for pearlitic grades.

The next group are INADVERTENT ALLOYS not necessarily poisonous, but as pearlite formers the can prevent meeting ferritic grades, as-cast.

Copper is added up to 1.0% in pearlitic high strength grades. But, in ferritic grades a residual of 0.10% copper can cause too much pearlite.

Tin likewise is sometimes added for pearlitic grades, but should be below 0.01% in ferritic grades.

Arsenic danger limits are 0.02% and 0.05% and molybdenum limits 0.03% and 0.30% for ferritic and pearlitic grades.
Table 2. Trace Elements Subversive to Graphite Nodularity

<table>
<thead>
<tr>
<th>Element</th>
<th>Max. safe limit</th>
</tr>
</thead>
<tbody>
<tr>
<td>Lead</td>
<td>.002%</td>
</tr>
<tr>
<td>Antimony</td>
<td>.002%</td>
</tr>
<tr>
<td>Bismuth</td>
<td>.002%</td>
</tr>
<tr>
<td>Titanium</td>
<td>.05%</td>
</tr>
<tr>
<td>Tellurium</td>
<td>.02%</td>
</tr>
<tr>
<td>Aluminum</td>
<td>.04%</td>
</tr>
<tr>
<td>Zirconium</td>
<td>.01%</td>
</tr>
<tr>
<td>Selenium</td>
<td>.01%</td>
</tr>
<tr>
<td>Zinc</td>
<td>being determined</td>
</tr>
</tbody>
</table>

Cerium can extend safe limit

Undesirable Carbide Formers

<table>
<thead>
<tr>
<th>Element</th>
<th>Ferritic</th>
<th>Pearlitic</th>
</tr>
</thead>
<tbody>
<tr>
<td>Boron</td>
<td>.002%</td>
<td>.002%</td>
</tr>
<tr>
<td>Chromium</td>
<td>.05%</td>
<td>.10%</td>
</tr>
<tr>
<td>Vanadium</td>
<td>.02%</td>
<td>.05%</td>
</tr>
</tbody>
</table>

Pearlite Formers

<table>
<thead>
<tr>
<th>Element</th>
<th>Ferritic</th>
<th>Pearlitic</th>
</tr>
</thead>
<tbody>
<tr>
<td>Copper</td>
<td></td>
<td></td>
</tr>
<tr>
<td>Tin</td>
<td></td>
<td></td>
</tr>
<tr>
<td>Arsenic</td>
<td></td>
<td></td>
</tr>
<tr>
<td>Molybdenum</td>
<td>Neutral – Add Strength and Hardenability</td>
<td></td>
</tr>
<tr>
<td>Nickel</td>
<td>.30%</td>
<td>1.00</td>
</tr>
<tr>
<td>Cobalt</td>
<td></td>
<td>rarely encountered</td>
</tr>
</tbody>
</table>

Some elements like Ni and Co, are essentially neutral, contributing some strength and hardenability but considerable trace concentrations can be tolerated before any trouble.

Choice of Charge Components

LOW PHOSPHORUS PIG IRON is the most desirable charge material but has the highest price. Pig iron is uniform in size and known composition with low P, and S, Mn, and trace elements. Sorelmetal is very low in Mn and residual elements.

Pig iron in the charge produces and iron with some residual nuclei which is easier to inoculate. Most foundries charge a proportion of pig iron to stabilize composition and dilute any unwanted elements like P, S, Mn, and tramp elements from steel scrap.

Some producers make good ductile iron with no pig iron but with close attention and more risk. They usually have pig iron available to be added whenever the undesirable elements are about to climb near critical limits.

FOUNDERY RETURNS should be melted in the proportion they are generated. If the casting yield is 70%, then 30% return gates, runners, and scrap castings are available for remelting.
Some foundries charge more pig iron and less returns on ferritic grades, and charge more returns and less pig iron on pearlitic grades. Total use of returns should match total generation.

**Cast Iron Scrap** used on gray iron at generally attractive prices, *should not be used in ductile iron* because of too high P and S and greater risk of tramp elements.

**Steel Scrap** is attractive for ductile iron because of low P and S contents. Steel scrap is usually available at an attractive price, after allowance for silicon addition and recarburizers. There are many varieties and grades of steel scrap obtainable at various prices based on quality and size. Scrap prices fluctuate with supply and demand.

Two general categories are **PROCESS SCRAP** directly from steel mill or forge shop trimming operations and **OBSOLETE SCRAP** consisting of worn out automobiles, refrigerators, machinery, bridges, railroad cars, etc.

**Process scrap** is more desirable because analysis is more dependable and range is known. Bushelling scrap trimmed from thin sheet metal is especially desirable for ferritic grades because of low manganese levels as well as low trace elements. But, availability is limited and at a premium price.

**Obsolete scrap** is more available and at lower prices. It can be used carefully in some proportions on pearlitic grades and heat treated products, with precautions and close attention. Axles, shafts, and gears should be avoided because they are generally alloyed.

Structural and plate classification, in earlier years, was more reliably free of alloys. But, in recent years, chromium has been added and Mn increased in some rust resisting grades called “Corten”. This alloyed plate cannot be recognized.

Sheet steel, usually bundled, may be plated with chromium or tin, galvanized with zinc or painted with lead based paint, all contributing undesirable trace elements.

“Black bundles”, supposedly free of plating, can be obtained at a higher price. Shredded auto bodies, refrigerators, etc. are melted by some large foundries in controlled proportions with close monitoring of tramp element contamination. It is more important that ductile iron foundries maintain good relations and communications with their scrap dealers. With full knowledge of the contamination risks, the foundry management must seek the lowest cost metal charge within safe quality requirements of their product.

In determining the lowest cost charge mix, risk factors must be evaluated. Also, yield factors must be a part of the calculation because some types of scrap contain more dirt or slag and thin scrap with more surface area causes more oxidation loss and lower metallic yield.

Computer programs are available for calculation of “least cost mixes” from AFS and other sources.

**Recarburizers** are a part of the metal charge in electric melting. For ductile iron, more expensive low sulfur carbon and graphite carburizers must be used. High sulfur carburizers, much lower in cost, can be used on gray iron. Electric melting foundries making gray iron and ductile iron must be sure that high sulfur carburizers never go into ductile iron. Some ductile iron has been ruined by this mistake.
MANY MELTING METHODS SUITABLE
Most types of melting furnaces have been used in melting base iron for ductile treatment.

Electric Coreless Induction Furnaces are used by the greatest number of foundries. Pollution control costs caused many small cupolas to be replaced with electric furnaces.

Coreless furnaces provide flexibility and positive control. Alternating two or more furnaces can provide almost continuous supply of iron. With several furnaces, two or three types of iron can be poured.

Line frequency furnaces usually maintain a molten bath making multiple taps down to 2/3 full before charging back. The second furnace then supplies metal while the first furnace is melting back. The second furnace then supplies metal while the first furnace is melting back.

Since line frequency inductions require starter blocks for slow melting of a cold charge, a molten bath is maintained and generally held overnight.

The newer medium frequency power sources can melt more for the furnace size, can be tapped lower and will melt cold charges faster. This increased flexibility makes it possible to melt out and come back with another grade of ductile iron or another type of iron.

Channel Induction Furnaces are very popular for holding furnaces. Many large cupolas are duplexed into large channel furnaces to level out demand and provide closer control of temperature and chemistry.

Some foundries use channel furnaces for slow melting at night with lower cost, off-peak power. Then on the day shift, the full furnace dispenses the metal through the operating shift. Where this type of melting is done, the vertical channel furnace is more open for cold charging than the barrel shaped channel.

A molten heel must be maintained making it difficult to change from gray iron to ductile iron or from pearlitic to ferritic ductile iron.

Electric Arc Furnaces are used by a few foundries. Flexibility for fractional taps is not as good as on the induction furnace. Bath uniformity is not as good and the added electrode cost usually makes arc melting costs higher.

The Cupola remains the favored melting furnace in large tonnage, high production foundries and pipe shops. Large cupolas can usually run a lower melting cost depending on relative costs of coke and electric power.

The Basic Slag Cupola was developed in the 1950’s to melt low sulfur iron for ductile iron. Depending on slag basicity level, cupola iron can be produced at very low sulfur levels and with high carbon pick up from high steel charges. These were favorable for ductile iron but Si loss much higher than the loss on the acid slag cupola was an economic disadvantage.

At one time, many cupolas were operated “basic” for ductile iron. But, with development of several external desulfurizing methods and increasing cost of Si, many found that
desulfurized acid cupola metal provided a lower total cost. A few foundries still melt in basic slag cupolas. But, the largest tonnage of ductile iron is melted in neutral or acid slag, water cooled cupolas, with external desulfurizing and usually duplexed into channel holding furnaces.

**The Cokeless Gas Fired Cupola** is used by some foundries in Europe producing ductile and gray iron. The cokeless cupola picks up no sulfur which is favorable but picks up no carbon and must be recarburized when melting steel scrap. Some of the most successful are duplexed into channel holders.

**MELTING CONTROLS OF BASE IRON**

Closer control of base iron composition requires closer melting controls. The P, S, Mn, and tramp elements are determined by choice and monitoring of charge materials. The required silicon and carbon ranges are adjusted and controlled in the melting operation.

C and Si are graphitizing elements which encourage graphite precipitation and prevent the formation of carbides.

The combined graphitizing effect of C and Si is reflected in carbon equivalent which in simples form is:

\[ CEq = C + \frac{1}{3} Si \]

Popular final analysis ranges for various thicknesses are:

<table>
<thead>
<tr>
<th></th>
<th>¼” thickness</th>
<th>½” – 2”</th>
<th>Over 4”</th>
</tr>
</thead>
<tbody>
<tr>
<td>C</td>
<td>3.65 – 3.80%</td>
<td>3.55 – 3.70%</td>
<td>3.40 – 3.60%</td>
</tr>
<tr>
<td>Si</td>
<td>2.50 – 2.80%</td>
<td>2.40 – 2.70%</td>
<td>2.30 – 2.60%</td>
</tr>
<tr>
<td>CEq</td>
<td>4.50 – 4.60%</td>
<td>4.40 – 4.50%</td>
<td>4.30 – 4.45%</td>
</tr>
</tbody>
</table>

(higher strength → lower CE%)

On very thin castings, with carbide tendency, it is advisable to target toward the higher ranges or higher. For heavy section castings, it is generally advisable to target toward the lower range.

Carbon flotation defect becomes a threat above 3.75% C and 4.55 CEq, but occurs more readily in heavy sections and hardly has time to occur in very thin sections. When low temperature impact applications make lower Si advisable or 2.50% max silicon is specified, then silicon range should be dropped to 2.20 – 2.50% and carbon increased about .10%.

These are final composition ranges. However, the treatment process usually lowers carbon to .10 to .15%. Si from the treatment alloy and the post inoculation can contribute 1.00 – 1.50% Si.

Consequently, in order to meet the final ranges suggested, for the ¼” in. section, the preliminary analysis tapped from the melting furnace could be:

<p>| | |</p>
<table>
<thead>
<tr>
<th></th>
<th></th>
</tr>
</thead>
<tbody>
<tr>
<td>C</td>
<td>3.75 – 3.90%</td>
</tr>
<tr>
<td>Si approx.</td>
<td>1.20 – 1.70%</td>
</tr>
<tr>
<td>CEq</td>
<td>4.25 – 4.45%</td>
</tr>
</tbody>
</table>
Each foundry should determine their furnace targets after allowance for carbon loss and silicon increase from their treatment and inoculation practices used.

In cupola melting, the analysis from the cupola is considered preliminary to be adjusted in the holder to desired final targets.

In electric furnace melting, a preliminary sample is taken at melt down. While being evaluated, the furnace is slagged off. Then, additions of C and/or Si are made to adjust to obtain final target ranges. One very useful instrument has been the eutectic arrest carbon equivalent instrument. These instruments follow the cooling curve of a solidifying sample and from the temperature of the liquidus and solidus arrests, the CE is determined with good accuracy, then C is calculated with fair accuracy and Si with less accuracy.

The carbon equivalent instruments have enabled foundries to quickly check composition on the furnace floor and make adjustments toward better final control.

Later refinements of these instruments by analyzing certain characteristics of the cooling curve can predict certain characteristics of the iron structure.

If a Spectrometer is available with fast reporting potential, a Spectrometer preliminary sample can be run to guide final adjustments.

**TEMPERATURE CONTROL IS ESSENTIAL**
A part of melting control is controlling the temperature consistently at which all operations are performed. Especially important are closely controlled treatment temperature and pouring temperature.

Thermocouples provide the most positive temperature reading with a digital read out. Optical and infrared pyrometers have been used but are more dependent on the skill of the operator and conditions of smoke and slag.

Tapping temperature is usually the treatment temperature, which influences magnesium recovery. The tap-treatment temperature then determines ultimate pouring temperature.

Ductile iron should be treated at the lowest temperature that will give the desirable pouring temperature. Some are able to treat at 2700F, others require 2800F and higher. All efforts should be made to minimize temperature loss by thorough preheating of ladles, and use of covered pouring ladles or Kaowool blankets. Inefficient temperature loss necessitates higher treatment temperature and the unnecessary cost of extra magnesium.

Pouring temperature affects solidification mechanism and likelihood of casting defects, and should be controlled within a range established by successful experience. More problems are usually encountered from pouring too cold.

Electric furnace iron should not be overheated or held for long periods at high temperature. Residual nuclei can be burned out making it more difficult to inoculate overheated iron.
Electric arc furnace iron is difficult to inoculate presumably because more residual nuclei are burned out under the high temperature arc. Floating graphite can reduce this tendency.

In coreless induction furnaces, second and third taps are sometimes more difficult to inoculate than first taps due to longer holding. Cupola iron is easier to inoculate presumably because contact with hot coke maintains some residual nuclei. But, cupola iron heated and held in electric holding furnaces can lose some of the residual nuclei.

Type of melting, charge material, temperature history, and holding time all influence residual nuclei and ease of inoculation. This makes it advisable to add enough inoculant to nucleate against all possible conditions.

**DESULFURIZING METHODS**

Several external desulfurizing methods are successfully used to remove sulfur to the low ratios needed for controlled treatment of ductile iron.

For effective desulfurization to low levels, the two essentials are a reagent that will absorb sulfur and secondly, a stirring action that will bring the iron continuously in contact with the desulfurizing material.

**DESULFURIZING REAGENTS**

Calcium carbide (CaC2) is the simplest and most effective desulfurizing material. Additions of 0.5% to 1.0% regularly desulfurize cupola iron as high as .10% sulfur to final levels of .01% sulfur. Some safety and environmental precautions must be followed in using carbide.

LIME AND FLUORSPAR mixtures are used effectively, especially in areas where environmental concerns prohibit carbide. The fluorspar (CaF2) must be carefully proportioned, usually about 1%-5%, of the lime (CaO). Controlling the proportions adds some complexity but overall cost of desulfurizing may be lower.

SODA ASH (Na2CO3) used for forehearth desulfurizing or gray iron is not effective for very low sulfur levels. Refractory attack is excessive and effectiveness is inadequate for .01% - .02% sulfur levels.

MAGNESIUM-LIME mixtures have been used in steel mill desulfurization.

**MIXING METHODS**

INJECTION was one of the early methods of stirring in desulfurizing agents. From a hopper, carbide was forced by gas pressure through a refractory or graphite lance, feeding the carbide into the iron and providing agitation for effective metal contact.

SHAKING LADLES have been used by some of the high production foundries.

Calcium carbide is fed into a ladle, cradled in a shaker mechanism that provides a reciprocal rotary stirring motion.

REFRACTORY STIRRER method uses a refractory paddle on a shaft driven by an electric motor. The stirrer is lowered into the iron providing an “egg beater” type of agitation while carbide is fed in. This method is more adaptable to large ladles and requires longer time.
POROUS PLUG mixing has become the most popular method, used by many large and small foundries.

Through one or more porous refractory plugs in the bottom of a ladle or special forehearth, inert gas, usually nitrogen, under pressure, passes through the metal and produces a swirling almost fountain action. Carbide is fed from an overhead hopper into the swirling action.

The porous plug can be used in the ladle for intermittent batch desulfurizing, where both gray iron and ductile iron is produced. Ductile iron is sometimes treated with nodulizing alloy in a porous plug ladle after desulfurizing with carbide.

The porous plug is also used effectively for continuous desulfurization in high tonnage cupola operations.

Iron from the cupola is by-passed into a refractory lined vessel or forehearth, outfitted with one or three porous plugs. The desulfurizing reagent (carbide or lime-spar) is fed continuously at a controlled rate onto the swirling surface. Clean, low sulfur iron flows continuously out the bottom over a dam and on to the holding furnace or forehearth.

Spent carbide spills over or is raked off the top into a container for safe disposal.

For consistent treatment control, sulfur should be controlled within the following ranges:

For ladle treatment .015% - .025%
For In-mold treatment .005% - .010%

GASEOUS CONTAMINATING ELEMENTS
Several gaseous type elements, in small concentrations, can contaminate irons and steels. Because of the high expectations of ductile iron, excessive trace concentrations must be understood and avoided. There is much yet to be learned about these strange contaminants.

IRON OXIDE RESIDUAL results from exposure to air, rust and moisture. In the cupola, a low bed can cause "overoxidized" iron.

In the electric furnaces, excessive scrap can cause high iron oxide residual. Iron oxide content can affect the liquidus arrest – temperature curve.

High iron oxide content consumes more magnesium, causing lower recovery and precipitating undesirable inclusions. Magnesium treatment reduces iron oxide content to lower and narrower ranges.

<table>
<thead>
<tr>
<th>Iron Oxide ranges</th>
<th>As Melted</th>
<th>After Mg Treatment</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td>0.006 - .013%</td>
<td>.001 - .008%</td>
</tr>
<tr>
<td></td>
<td>60 – 130 parts per million</td>
<td>10 – 80 parts per million</td>
</tr>
</tbody>
</table>

After treatment, ductile iron can be reoxidized by oxidizing slag buildup in ladles, back iron shot, excessive turbulence in pouring and gating, and from moisture from new ladle linings or patches not thoroughly heated. Molding sand with high moisture and low sea coal can contribute iron oxide and hydrogen.

NITROGEN is present in irons at various residual levels and can be a hidden “evil spirit”.
Molecular nitrogen gas is not absorbed into iron. But, dissociated atomic nitrogen under the electrodes of the electric arc furnace is absorbed. And nitrogen is contributed by some chemical no-bake binders and from some alloys. Nitrogen can be analyzed by a proficient chemical laboratory.

<table>
<thead>
<tr>
<th></th>
<th>As Melted</th>
<th>After Mg Treatment</th>
</tr>
</thead>
<tbody>
<tr>
<td>Nitrogen ranges</td>
<td>0.008 - .0150%</td>
<td>.001 - .008%</td>
</tr>
<tr>
<td></td>
<td>80 – 150 parts per million</td>
<td>10 – 80 parts per million</td>
</tr>
</tbody>
</table>

The violence of magnesium treatment flushes out much or the nitrogen as melted. But, treated iron containing magnesium more readily absorbs any nitrogen exposed from the mold and cores.

Nitrogen at low concentrations stabilizes pearlite and contributes some strength.

At higher concentrations, nitrogen increases carbide tendencies. At very high concentrations, excess nitrogen can be expelled upon solidification causing gas holes.

Nitrogen trouble is seldom encountered below 40 parts per million, but problems are likely above 100 parts per million.

HYDROGEN can have hidden strange effects on iron and steel. In cast and wrought steels, high hydrogen content causes embrittlement and fine cracks in forging. Hydrogen escapes during and after solidification and is difficult to contain and analyze.

Hydrogen is absorbed in melting from moisture in leaking tuyeres air, damp charge materials, wet alloys, and insufficiently heated ladle linings or patches.

<table>
<thead>
<tr>
<th></th>
<th>As Melted</th>
<th>After Mg Treatment</th>
</tr>
</thead>
<tbody>
<tr>
<td>Hydrogen ranges</td>
<td>0.0002 - .0015%</td>
<td>.0002 - .0004%</td>
</tr>
<tr>
<td></td>
<td>2 – 15 parts per million</td>
<td>2 – 4 parts per million</td>
</tr>
</tbody>
</table>

Magnesium treatment alsoflushes some of the hydrogen absorbed in melting. But, after treatment, the magnesium in the iron greatly increases hunger for hydrogen from the pouring ladles or molds.

Hydrogen is readily absorbed from:
- damp alloys
- green troughs, ladle linings and patches
- high moisture, low sea coal sand

Hydrogen first increases carbide tendency. Hydrogen diffuses toward the center of casting sections where it can cause inverse chill or center line carbides.

High concentrations of hydrogen can be expelled upon solidification causing gas porosity or “pin holes”. Hydrogen troubles can be experienced with concentrations or only four parts per million.

HIGH SULFUR IN SAND AND FACINGS, though not a “gaseous element “, can mysteriously contaminate and cause flake graphite on the surface of ductile iron castings.

In sand with sulfur buildup to 0.2%, flake graphite has occurred for some depth in the skin of ductile iron castings.
It is recommended that sand be analyzed for sulfur and kept below 0.15% max. Sulfur by increasing new sand additions. Some foreign sea coal has been found to contain as much as 5.0% sulfur and some facings are dangerously high in sulfur.

Nitrogen in combination with sulfur, increases flake tendency more readily.

**Complete control or ductile iron composition must include understanding and control of these strange contaminating influences - iron oxide, nitrogen, hydrogen and mold sulfur.**

### CHAPTER 4

**TREATMENT METHODS AND CONTROLS**

**NODULIZING ALLOYS**

Base iron melted to controlled chemistry and temperature and desulfurized, if required, is ready for magnesium nodulizing treatment.

Pure magnesium is not practical for iron addition without special equipment. Because of its volatility and reactivity, recovery would be less than 10% and uncontrollable. Treatment with pure magnesium requires special equipment to plunge, envelope, or pressurize.

For more practical use, magnesium is alloyed to reduce violence and reactivity.

**NICKEL MAGNESIUM ALLOYS**

The first alloys were nickel magnesium alloys developed by International Nickel Company, the inventor and licensor of ductile iron.

Without special equipment, nickel magnesium alloys can be thrown into a ladle with reasonable recovery of 50% to 70% and are easier to control. However, nickel alloying is more expensive and nickel interferes with obtaining high ductility ferritic grades as-cast. Nickel contributes strength and can be beneficial in pearlitic grades.

Nickel magnesium alloys are ideally suited for the treatment of special austenitic, and acicular ductile irons which utilize the alloying benefits of nickel.

Many small foundries, with limited technical staff and equipment, are producing some ductile iron using nickel magnesium alloys for ease of treatment and more positive control.

Following are compositions of typical nickel magnesium alloys available:

<table>
<thead>
<tr>
<th>INCOMAG ALLOYS</th>
<th>Mg</th>
<th>Si</th>
<th>C</th>
<th>Fe</th>
<th>Ni</th>
</tr>
</thead>
<tbody>
<tr>
<td>#1</td>
<td>13</td>
<td>16</td>
<td>2.0 max</td>
<td>5.0 max</td>
<td>80%</td>
</tr>
<tr>
<td>#2</td>
<td>13</td>
<td>16</td>
<td>2.0 max</td>
<td>5.0 max</td>
<td>50%</td>
</tr>
<tr>
<td>#3</td>
<td>4.2 – 4.8%</td>
<td>30%</td>
<td>2.0 max</td>
<td>35%</td>
<td>95%</td>
</tr>
<tr>
<td>#4</td>
<td>4.0 – 4.8%</td>
<td>30%</td>
<td>2.5 max</td>
<td>35%</td>
<td>60%</td>
</tr>
</tbody>
</table>

Because of density greater than iron, small piglets of nickel magnesium alloys can be tossed into a ladle of treated iron where indications suggest insufficient magnesium recovery from regular treatment.
MAGNESIUM FERROSILICON ALLOYS

Alloys of magnesium with ferrosilicon have become most treatment materials. Mg recovery is not as good as with nickel magnesium, but since the silicon is utilized in final composition, alloy cost is lower. High ductility grades can be produced as cast. As shown in table 3, magnesium ferrosilicon alloys are available at several levels of magnesium which may be preferred for various treatment methods.

The most popular magnesium levels are 3%, 5% and 9%. No attempt is made to list all the alloy combinations of all alloy producers. Within each magnesium level, several combinations of cerium and other trace elements are available.

Following is a condensed summarization of typical magnesium ferro-silicon alloys used.

Table 3. Typical Mg Ferrosilicon Alloys

<table>
<thead>
<tr>
<th>Alloy Designation</th>
<th>Chemical composition %</th>
<th></th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td>Mg</td>
<td>Si</td>
</tr>
<tr>
<td>3% MgFeSi</td>
<td>2.9 - 3.5</td>
<td>46</td>
</tr>
<tr>
<td>5% MgFeSi</td>
<td>5.0 - 6.5</td>
<td>46</td>
</tr>
<tr>
<td></td>
<td>5.0 - 6.0</td>
<td>46</td>
</tr>
<tr>
<td></td>
<td>5.0 - 6.0</td>
<td>46</td>
</tr>
<tr>
<td></td>
<td>5.0 - 6.0</td>
<td>46</td>
</tr>
<tr>
<td>9% MgFeSi</td>
<td>8.5 - 10</td>
<td>45</td>
</tr>
<tr>
<td></td>
<td>8.5 - 10</td>
<td>45</td>
</tr>
<tr>
<td></td>
<td>8.5 - 10</td>
<td>45</td>
</tr>
</tbody>
</table>

Various special grades are available containing either barium, higher calcium, higher lanthanum, or other special trace elements.

Cerium free grades are available, as well as several levels of cerium, incorporated in the alloy. The most widely used alloys contain 1% cerium or 1% total rare earths. Cerium and the other rare earth elements, in addition to counteracting tramp elements, also help magnesium to dioxide, desulfurize and nodulize. Excessive cerium increases carbide tendency.

Special benefits are claimed from certain trace elements in the alloy.

CALCIUM reportedly reduces violence and improves performance. Normal level of 1% is increased to 1.5% and 2% calcium levels in special alloys available.

Benefits are claimed for barium at concentrations of 1% to 3% in magnesium ferrosilicon alloys, in certain circumstances.

LANTHANUM enriched alloys with a higher ratio of lanthanum to cerium reportedly give special benefits under certain conditions.

Various alloy companies in various countries offer their alloys with specific composition ranges of important elements. New alloys are continually being developed with special compositions.

Most alloy producers provide complete analysis of each lot of their alloys and use SPC programs to assure close control. Alloy analyses should be a part or the quality control file.
Nodularity failures have been encountered where a purchasing department got a low price deal from a new alloy supplier. However, the new alloy was lower in cerium and favorable trace elements, resulting in lower recovery and low nodularity. Some alloys are more oxidized than others.

When treatment alloy is obtained from a new source, first ladles should be checked closely. Any new alloy lot should be tried before all the previous alloy lot has been depleted.

Even a new lot or alloy, from the same source, can have lower magnesium and cerium within the specified ranges and yield lower magnesium residuals if alloy weight is not adjusted.

**CERIUM - RARE EARTH ALLOYS**

Nickel magnesium alloys and pure magnesium, when used for treatment, contain no cerium. If cerium is desired to minimize the detrimental influence of subversive elements, it can be added from several rare earth alloys available, like mischmetal.

Cerium is the major active element. But, other elements in the rare earth family like lanthanum, praseodymium and neodymium assist cerium.

<table>
<thead>
<tr>
<th>Typical Rare Earth Alloys</th>
</tr>
</thead>
<tbody>
<tr>
<td>High CE</td>
</tr>
<tr>
<td>Cerium: 92%</td>
</tr>
<tr>
<td>Lanthanum: 5%</td>
</tr>
<tr>
<td>Praseodymium: 1%</td>
</tr>
<tr>
<td>Neodymium: 2%</td>
</tr>
<tr>
<td>Other CE: Bal.</td>
</tr>
<tr>
<td>Mischmetal: 48.5%</td>
</tr>
<tr>
<td>Cerium: 5%</td>
</tr>
<tr>
<td>Lanthanum: 28.8%</td>
</tr>
<tr>
<td>Praseodymium: 5.2%</td>
</tr>
<tr>
<td>Neodymium: 12.5%</td>
</tr>
<tr>
<td>Other CE: Bal.</td>
</tr>
</tbody>
</table>

In addition, RARE EARTH SILICIDES have been cerium and rare earths. Following are two rare earth silicides.

<table>
<thead>
<tr>
<th>Rare Earth Silicide</th>
<th>Total R.E.</th>
<th>CE</th>
<th>Si</th>
<th>Ca</th>
<th>Al</th>
<th>Fe</th>
</tr>
</thead>
<tbody>
<tr>
<td>A</td>
<td>30 – 35%</td>
<td>15 – 18%</td>
<td>30%</td>
<td>1%</td>
<td>1%</td>
<td>Bal.</td>
</tr>
<tr>
<td>B</td>
<td>10 -15%</td>
<td>9 – 11%</td>
<td>38%</td>
<td>4%</td>
<td>0.8%</td>
<td>Bal.</td>
</tr>
</tbody>
</table>

**ALLOY SIZE CONTROL AFFECTS RECOVERY**

The size or the treatment alloy affects its reactivity and recovery. Different size ranges are recommended for different treatment processes. The alloy supplier can recommend the best size range for the treatment method and conditions of use.

<table>
<thead>
<tr>
<th>Typical Size Ranges of Mg Alloys</th>
</tr>
</thead>
<tbody>
<tr>
<td>Treatment Methods</td>
</tr>
<tr>
<td>Open Ladle, sandwich, tundish</td>
</tr>
<tr>
<td>Flotret</td>
</tr>
<tr>
<td>In-mold</td>
</tr>
</tbody>
</table>

Excessive fines can lower magnesium recovery in most treatment processes. "Fines" tend to accumulate in the bottom of a container. Treatment with excessive fines from the bottom of a container has caused low nodularity ladles.

It is recommended that the last finer alloy in the bottom of a container, be placed on top of the new container for better size distribution.
NODULIZING TREATMENT METHODS
Many treatment methods have been developed and used to mechanically assist the absorption of magnesium into iron. (6)

Some methods, popular for awhile, have been virtually abandoned as other methods have been developed. It can be expected that more treatment methods will be invented in the future.

A full description of details of all treatment methods is not intended in this guide. A brief description or the more popular methods is presented to assist in selection of the most suitable process and awareness of the variables that must be controlled.

OPEN LADLE OR POUR OVER method, shown in Fig. 3, is the simplest and most flexible, especially for jobbing foundries with intermittent production or ductile iron. A weighed amount or treatment alloy is added to the bottom or a treatment ladle, and a weighed volume of iron is poured as quickly as possible over the alloy, ideally 15 seconds per ton.

With denser nickel magnesium alloys, reasonable recovery or 50 to 70% may be realized. But with magnesium ferro silicon alloys, recovery as low as 20% to 30% is likely. Ladles should be deep, with height twice or more the diameter to give greater depth of iron over the alloy. A pocket to contain the alloy on the opposite side from stream impingement delays ignition or the alloy until some iron has flowed back over the alloy.

SANDWICH TREATMENT is a refinement of the pour over method utilizing a protective cover of steel punchings, ductile borings, or ferro silicon over the alloy as shown in Fig 4. The cover delays reaction with the alloy until considerable iron depth is obtained. A steady consistent pouring rate is essential.

Some foundries note and record time to fill the ladle and time until magnesium flashes as indication or consistent mechanics in this process. With a good pocket, good cover and controlled pouring, recoveries may be improved to 40% or even 50%. But, rusty or damp cover steel has been observed to consume more magnesium than saved by the protection or the cover, causing low nodularity.

THE COVERED TUNDISH LADLE uses a cover as shown in Fig 5. Iron is poured into a tundish basin and through an orifice which controls the rate of pour and directs the stream to the side opposite the alloy pocket.

The ladle cover encloses the magnesium vapor flare and smoke. Some tundish covers are attached and some simply sit on the ladle and are lifted off after treatment. If slag buildup is controlled and the orifice and pocket are maintained, magnesium recoveries as high as 60% and higher have been experienced along with desirable environmental improvement.

Several treatment methods can be considered “TRICKLE IN” methods in that fine alloy is fed slowly into the iron to obtain practical recovery.
Fig 3. Open ladle-pour over method

Fig 4. Sandwich treatment method

Fig 5. Tundish ladle treatment

Fig 6. Porous Plug Method
INJECTION was an early method in which fine alloy or magnesium powder was driven by gas pressure through a graphite lance into the iron. Injection has been essentially abandoned in favor of simpler methods.

THE T-NOCK PROCESS, in Europe, discharged the iron through a simple refractory funnel in the bottom or a trough. Fine magnesium alloy was fed from a hopper above into the metal vortex.

POROUS PLUG TREATMENT has been the most popular of the "trickle in" methods. As shown in Fig. 6, inert gas is passed through a porous refractory plug in the bottom of the ladle, creating a swirling stirring action. Treatment alloy is fed into the middle of the turbulence. The gradual feed rate goes into solution with about 30% recovery.

The same porous plug system is used effectively for desulfurizing as previously described. In some foundries this equipment first desulfurizes with carbide added. Then after slagging off, magnesium alloy is added for treatment.

A WIRE FEED METHOD has been developed in recent years. Magnesium alloy inside a hollow wire is uncoiled and fed into the iron at a controlled rate. The same equipment is used to feed inoculants.

THE PLUNGING METHOD improved magnesium recovery by forcing the magnesium alloy into the iron as shown in Fig. 7. Alloy in a can is pinned into an inverted refractory bell at a plunging station. A ladle of iron is transferred to the station where a hydraulic cylinder forces the bell and alloy deep into the iron as a cover closes over the top of the ladle. Holes in the bell provide gradual contact of iron with alloy. Plunging made possible the use of higher magnesium alloys, magnesium impregnated coke, and briquets of magnesium turnings. Disadvantages are the need to travel to special equipment and possibility of considerable temperature loss if bell is not kept hot with fast use.

Several methods can be described as FLOW THROUGH METHODS in which metal flows through treatment alloy contained in a pocket.

THE FLOTRET method, shown in Fig. 8, utilizes a special trough with a pocket to contain a weighed quantity or magnesium alloy, usually 3.5% or 5% magnesium alloy. A refractory knife and dam control the flow. Treated iron leaves the trough. Magnesium recoveries of 60%-70% are reported.

Sometimes this process can treat directly into the pouring ladle eliminating one reladling. Post inoculant can be added into the stream from the trough.
Good maintenance or the special trough is required to maintain proper balance of inlet and outlet hole size. Keeping the box full and maintaining steady pouring during the reaction is very important.

IN-MOLD TREATMENT utilizes the flow through principle but inside the mold.

In this process, a chamber is formed in the mold where it is a part of the pressurized gating system. As the mold drag passes down the molding line, as shown in Fig 9., a weighed amount of 5M X 18M 3.5% magnesium alloy is placed in the chamber or pocket. When the mold is poured, iron flows through the alloy in the chamber retaining the right concentration or magnesium for good nodularity.

Smoke and reaction fumes are contained in the mold which is an environmental advantage.
Nodularity control depends on proper dimensions and proportions of all elements of the gating system. Since this requires much trial and error fine tuning, the in-mold treatment is more suitable for high production repetitive production and is not suitable for a jobbing variety of castings.

In-mold treatment requires very low sulfur base iron of 0.010% maximum sulfur and requires desulfurization, even with electric melting. Mg recovery is 70% - 80%.

In-mold iron is well nucleated just before solidification, producing fine nodules with a high nodule count and freedom from carbides. Inclusions tend to be higher. Casting yield is reduced by the chamber and gating modifications.

Precautions must be taken to be sure no mold is missed. If a mold is bad and will not be poured, the alloy must be removed to prevent its contaminating the sand system.

Nodularity verification is more complex. A simple micro cannot verify nodularity or many castings from an entire ladle since each mold is a different treatment batch. Automatic detection and vacuum removal systems are used to protect against this possibility.

Usual practice is to test each casting with ultrasonic or resonant frequency instruments in a finishing line. Calibration of these instruments is based on micros with satisfactory nodularity.

Also, in-process micros are taken from runners or knock-off lugs at established intervals to insure good process control and satisfactory microstructure.

The in-mold process uses a special reaction chamber built into the gating system. The molten iron flows over the Mg alloy to maximize Mg recovery while minimizing reaction flare, smoke and fumes.

Fig 9. In-mold treatment process
The converter process uses pure Mg inserted into a special compartment isolated from the molten metal until the ladle is filled and rotated from the horizontal to the vertical position

Fig 10. Fischer converter treatment

In recent years, the use or in-mold treatment has increased among the high volume producers, especially automotive foundries.

PURE MAGNESIUM PROCESSES
Several treatment processes use pure magnesium in specialized equipment. Low treatment costs can be realized if the volume justifies the cost or equipment and complex maintenance.

THE CONVERTER is a licensed process used by a number of foundries around the world.

In a special vessel, when rotated to the horizontal position, pure magnesium is inserted into a chamber in the upper corner with holes of controlled dimension, as shown in Fig. 10. The chamber opening is then closed, and the vessel is filled with a controlled weight of iron. When rotated into the vertical position, the magnesium in the chamber, now at the bottom, percolates up through the iron.

The converter in some applications has treated high sulfur iron successfully but more inclusions are possible. Mg recovery is 50% - 60% and cycle time is 7 to 12 minutes.

A MODIFIED CONVERTER has attracted some recent interest because it can treat smaller quantities or iron within a shorter cycle time and can be fitted into existing handling systems.

A tundish type ladle has a chamber added to the bottom with vertical holes connecting into the ladle. A bottom plate swings open for adding pure magnesium into the chamber, after which the bottom cover is clamped against a paste board seal.

When iron is poured through the tundish into the ladle, magnesium vapor passes through the holes and treats the iron. Small quantities of 2000-3000 lbs have been treated and handled within the existing monorail system.

Advantages or treating with pure magnesium are:

- lower treatment cost
- silicon flexibility to use more return or layer cost or silicon source
- ability to treat higher sulfur iron
- less carbon loss and need for rectorburizer
A PRESSURE LADLE is used by a few producers in the U.S. and Japan. Iron is poured into a specially constructed heavy ladle which is sealed and pressurized to 15 to 40 psi. Pure magnesium billets are then plunged into the iron. The imposed pressure forces more magnesium into the iron. Usually the iron is overtreated 3 to 4 times and blended with untreated iron to net the magnesium concentration desired.

REFRACTORY COATED MAGNESIUM BILLETS are plunged in a process developed in France. The refractory coating delays the magnesium reaction until fully submerged.

When treating with pure magnesium, more post inoculation is usually required.

From whatever treatment method is chosen, best quality control and product uniformity requires control of:
- Magnesium within a .015% range
- Cerium within a .005% range

For thin castings from low base sulfur iron
- Mg range of 0.03% - 0.045% is usually best

For heavy sections or from high sulfur base iron
- Mg range of 0.040% - 0.055% may be needed to insure good nodularity

TREATMENT VARIABLES AND CONTROL
All treatment processes are subject to many variables which must be understood and controlled. These include iron variables, alloy variables, and mechanical variables of the treatment process used.

IRON VARIABLES
- In every process, IRON TEMPERATURE affects magnesium recovery. Recovery decreases with increased iron temperature. Intentional or accidental temperature increases can cause low nodularity if magnesium alloy is not increased.
- Higher BASE SULFUR requires more magnesium addition. Unrecognized sulfur increases have caused low nodularity.
- More oxidized iron requires more magnesium to first deoxidize.

ALLOY VARIABLES
- Size variations can cause variations in recovery.
- Cerium content variation between sources or lots can affect nodularity.
- Variations in the trace elements can influence alloy recovery and effectiveness.
- Different lots from the same supplier and specification can vary 0.5% in magnesium or .20% in cerium requiring minor adjustments with change or lots.

MECHANICAL VARIABLES
All treatment processes have mechanical features to help magnesium recovery. Variations in these critical features can cause missed nodularity. Technicians should be trained to understand and control these variations.
For example, some potential variations in SANDWICH TREATMENT are:

- Ladle depth. Pocket condition.
- Rusty cover steel. Loose cover. Poorly placed.
- Irregular interrupted pour.
- Over poured, excessive iron weight.
- Excessive cover
- Slag build up

In the FLOW THROUGH METHOD, critical dimensions or the trough must be maintained, keeping the dimensions of the inlet and outlet controlled for proper low rate. Deterioration of the trough dimensions can cause low nodularity.

In the TUNDISH LADLE method, slag build up must be controlled and orifice size maintained.

POST INOCULATING ALLOYS AND METHODS
The second step, after magnesium treatment, is post inoculation with ferrosilicon which nucleates or triggers graphitization, preventing the formation of carbides. Without sufficient inoculation, excessive carbides tend to form which are very detrimental to ductility. Carbides are most likely in the corners of thin sections.

Since this nucleating effect fades with time, the inoculant should be added as near pouring time as possible.

In most foundries, the inoculant is added into the stream while pouring from the treatment ladle into the pouring ladle. The ladle is quickly slagged and poured within a few minutes.

Some of the best controlled foundries use a timer that sounds an alarm after 8 or 10 minutes, after which any remaining iron is pigged.

INOCULANT MATERIALS
The most widely used ladle inoculant is foundry grade 75% ferrosilicon. Calcium and aluminum residuals around 1% are necessary to the nucleating process. Higher calcium contents are recommended by some alloy manufacturers for more effective inoculation.

Some special inoculants, containing barium, reportedly experience longer fade time and more effective inoculation under certain conditions.

Inoculant size affects its solution and performance. Best size range can be recommended by the alloy manufacturer for the conditions involved.

Following are size ranges generally used:

<table>
<thead>
<tr>
<th>Condition</th>
<th>Size Range</th>
</tr>
</thead>
<tbody>
<tr>
<td>Large ladles</td>
<td>3/8” X 12 mesh</td>
</tr>
<tr>
<td>Medium ladles</td>
<td>1/4” X 20 mesh</td>
</tr>
<tr>
<td>Small ladles</td>
<td>5 mesh X 30 mesh</td>
</tr>
<tr>
<td>In stream injection</td>
<td>20 mesh X 80</td>
</tr>
<tr>
<td></td>
<td>30 mesh X 100 mesh</td>
</tr>
</tbody>
</table>

Excessive fines have caused inadequate inoculation and carbides.
Ductile Iron Society Research on Carbides and Inoculation

Twenty years ago, Research Report #4 provided some early guidance on factors influencing carbides and effectiveness of post inoculation.

Step bars, with six sections, from 1/8 in. to 2 in., were cast varying iron analysis, temperature, amount of inoculant and type of inoculant.

- Section size proved very sensitive. Inoculation practices for 1/2” section and even 1/4” sections gave excessive carbides in 1/8” section.
- Amount of inoculant showed a major effect. With 0.4% addition of 75% ferro silicon (.30% silicon) thicker sections were generally carbide free but, 1/8” sections had 5% to 20% carbides. A .9% addition (.75% silicon) was necessary to prevent carbides in the 1/8” section.
- Nodule count increased with better inoculation that eliminated carbides.
- Carbon equivalents near 4.6% were generally free of carbides in 1/8” sections but considerable carbides were experienced with 4.3% CEquivalent.
- Magnesium residuals .03 to .045% showed least carbides. Magnesium above .06% showed increased carbides and magnesium levels below .03% showed nodularity decline. Controlled cerium content is also important.
- Low pouring temperatures showed increased carbides over high pouring temperatures.
- Fading of inoculation occurs with time. After too long pouring time, a normally adequate inoculation can fade too much and produce carbides in thin sections.
- Faded inoculation can be easily restored with a small booster addition of FeSi in the sprue or mold, either granular tablets or solid inserts.
- Inoculating materials studied included six special inoculants with various traces or either strontium, barium, zirconium or high calcium. In some conditions, some or the special inoculants appeared to improve effectiveness. But, under most conditions, regular inoculating grade of 75% ferrosilicon (with proper calcium and aluminum residuals) appeared to be generally as effective. Other variables appeared generally more influential than the special inoculants.

MECHANICS OF INOCULANT ADDITION

In the typical foundry, with ladle treatment and with manual pouring, the best place to add the ferrosilicon inoculant is into the pouring stream from the treatment ladle into the pouring ladle. A cup on a long handle facilitates a smooth flow well directed into the stream throughout the pour, with most of the silicon recovered into the iron.

Poor recovery and effectiveness is experienced when the ferrosilicon inoculant is simply dumped into an empty ladle. Some silicon is oxidized and some imbedded in the slag with half or less going into solution.

Equipment is available which is attached to the ladle and feeds the ferrosilicon into the stream at a controlled rate and amount.

IN-MOLD BOOSTER INOCULATION

In larger molds, pouring time and solidification time is more likely to be longer after ladle inoculation and experience inoculation fade.

A small addition or granular ferrosilicon, in the sprue, will restore the nucleation, but uniformity and cleanliness may be a problem.
Mold inserts are more uniform and cleaner if properly used. Special blocks of ferrosilicon cast to various sizes and shapes are anchored in the runner. The ferrosilicon is dissolved evenly during the pour, restoring inoculation to maximum effectiveness.

Also, ferrosilicon tablets can be added with similar success.

STREAM ADDITION EQUIPMENT
In automatic pouring from a holding ladle or from manual stopper rod pouring, adding fine inoculant into the pouring stream is important. Several types of equipment are available.

AUTOMATIC INJECTION or spraying of fine ferrosilicon into the stream is widely used. The spraying of ferrosilicon is automatically activated when a stream of iron appears.

In the WIRE FEED METHOD, hollow wire filled with ferrosilicon is fed at a controlled rate into the stream or tundish basin.

A ROTATING PIPE METHOD developed by B.C.I.R.A., feeds fine inoculant from a rotating inclined pipe. The amount added is controlled by the speed of rotation and inclination of the pipe.

Inoculation effectiveness is much better when added in the stream into the mold. Late mold stream additions of .10% to .20% have been found as effective as pouring ladle additions of 0.50% to 0.80%.

With automatic stream injection devices thorough maintenance is important to be sure molds are not missed due to malfunction of the dispenser. In some cases, 2 or 3 injectors are used to minimize the risk of a malfunction.

Some of the most effective nucleation has resulted when basic inoculant is added into the transfer or pouring ladle, and then given late booster inoculation into the mold by one of the automatic feeder devices.

CHAPTER 5

NODULARITY CONFIRMATION BY MICROSCOPE

To assure quality in ductile iron, the most important test is a microstructural evaluation immediately after pouring each ladle. The microscope should be located near the pouring floor.

GENERAL MICROSCOPE PRACTICE
A small specimen is poured which can be quickly polished and percent nodularity determined. The microscope test should be poured from the last iron from the ladle which represents the most faded inoculation and magnesium content, the worst possibility from the ladle.

If nodularity is too low or carbides are present, decision must be made quickly to separate the castings from the questionable ladle before mixing in the shakeout and finishing operations. The held castings are usually "red tagged" and "quarantined" until final decision has been made, whether to accept, reject or reclaim.
Someone with adequate training in ductile iron metallurgy must be authorized to make final decisions.

When nodularity on the quick specimen is marginal or low, it should be verified by a micro sample from a runner or casting. Small specimens usually run 5% higher nodularity than the thicker castings. On the other hand, the sample specimen could be contaminated giving a false low rating.

For most applications, a minimum of 80% nodularity (Types I and II) in the casting is considered minimum to meet the tests and expectations of ductile iron. Generally, a minimum of 85% nodularity is required in the specimen to insure 80% in the casting. For safety parts, 90% nodularity is required.

MICROSCOPE EQUIPMENT
The basic control of nodularity and matrix required for good ductile iron can be accomplished with a simple table microscope with capacity for 100X magnification. Fig. 11 shows the simplest microscope and polishing equipment for quick examination.

Most high volume producers have a second more, versatile microscope in the laboratory with capacity for high magnifications and photomicrographs for the record or for customers when requested.

Some use a more complex IMAGE ANALYZING MICROSCOPE for quantitative metallography of cast irons. In addition to percentage nodularity, the analyzer can determine a graphite shape factor, nodule count, and proportion of pearlite.

Fig 11. Station For Quick Micro Examination

![Fig 11. Station For Quick Micro Examination](image)

Fig 12. AFS Test Coupon For Microexamination of Ductile Iron

![Fig 12. AFS Test Coupon For Microexamination of Ductile Iron](image)
MICROSCOPE SPECIMENS
Several types of specimens are used for quick microscope evaluation.

Many foundries use a TRIANGULAR WEDGE from which a slice is cut after breaking. One advantage of the wedge is immediate indication of any carbides on the tip, sometimes visible in the fracture and readily apparent after etching the polished specimen.

Some cast a BAR which can be bent to give early indication of ductility, then a slice is sawed or cut for polishing.

Some foundries with Disa vertically parted molds cast a lug or wafer onto the casting or runner positioned near the outer edge.

A technician, with pliers or tongs, punches through the sand and twists off the specimen, which is a good size for polishing. Such a specimen represents the iron in the mold after any reaction with the sand.

A popular specimen is the AMERIGAN FOUNDRYMENS SOCIETY SPECIMEN developed by Committee 5K as shown in Fig. 12.

Lugs of 3/8”, ½” or ¾” sections project from a basin about 1 ½” X 2 ½”, which gives the convenient sized specimens a cooling rate more comparable to castings of greater thickness.

Specimens are cut to size with an abrasive wheel or power saw

POLISHING EQUIPMENT AND PROCEDURE
The cut specimen is first rough ground on a belt or rotary sander.

Then, on the 4 faces of a disc polisher, polishing can progress over progressively finer papers.

A popular combination of grit sizes are:

<table>
<thead>
<tr>
<th>Silicon Carbide</th>
<th>Emery Paper</th>
</tr>
</thead>
<tbody>
<tr>
<td>180 grit</td>
<td>Fine Emery paper</td>
</tr>
<tr>
<td>280 grit</td>
<td>#1</td>
</tr>
<tr>
<td>400 grit</td>
<td>#1/0</td>
</tr>
<tr>
<td>600 grit</td>
<td>#3/0</td>
</tr>
</tbody>
</table>

A fair evaluation of nodularity can be obtained after the 600 paper. But, better delineation of graphite shape can be obtained by finish polishing on a rotary polishing wheel with an aqueous suspension of alumina applied to a low nap covered cloth wheel.

Newer diamond abrasives are suspended in a paste applied to the wheel with an applicator. Automatic polishing equipment is available where a high volume of metallography justifies. The best delineation of graphite shape can be obtained by etching lightly and then polishing off. A popular etchant is 2 % nital made with 2% nitric acid in 98% ethyl alcohol. The presence of any carbides can be detected after etching, with 2% nital. Fig. 13 shows specimens with 2% and 10% carbides. (5) Carbides appear as a white acicular needle like constituent. Ammonium persulfate is a specialized etchant that shows carbides in sharper contrast.
The time urgency is to insure acceptable nodularity and freedom from carbides. Visual comparator charts are necessary to evaluate nodularity percent accurately. A Ductile Iron Microstructures Rating Chart by AFS (No. C+18201) is sold by AFS. This wall chart portrays various levels of nodularity, carbides, pearlite, and nodule count.

Fig. 14 show nodularity ratings of 100%, 90%, 80%, and 70% from AFS "Guide to Ductile Iron Microstructures" (5).

Some foundries, with close control, make their own homemade standards with more intermediate increments of nodularity. More standards make it easier to rate marginal levels, or detect small differences.

Some indication of pearlite trend in the matrix can be determined on the etched specimen. However, if the specimen is smaller in section, the pearlite is likely to run higher than the tensile bar and the castings.

The microstructure of the slower cooled AFS specimen may more closely match the structure of test bars and castings.

A quality foundry should determine structural differences between their specimen, tensile bar, and various casting sections.

NODULARITY RECORDS
Since percent nodularity is so important to the mechanical properties of ductile iron, a good record should be maintained or nodularity rating with identification that can be traced to casting lots.

Fig 13. Carbides in the microstructure
Ideally, nodularity should be recorded by percentage with comparator charts available to assist. Some companies use ratings of 1, 2, 3, 4 or A, B, C, D to represent levels of nodularity.

The record should include rechecks of castings and disposition of any marginal or low batches.

**HOLDING MECHANICS**  
**ON CONTINUOUS CONVEYOR LINE**  
Nodularity evaluation is more urgent on a conveyorized molding line where poured castings are moving toward shakeout and cleaning operations. Any holding decisions need to be made usually within about 30 minutes, after polishing and evaluating a specimen from the last mold of the ladle.

The last mold from each ladle needs to be marked. Some insert a numbered metal tag into the last mold, others mark with white powder.
Whenever a ladle shows low nodularity or carbides, the castings from that ladle must be separated at some point.

Some foundries manually pull out the questionable castings into boxes ahead of the shakeout.

Some foundries have a mechanized by-pass shuttle which can "side track" castings from questionable ladles onto a by-pass conveyor where they can be isolated and quarantined until final decision is made.

ON FLOOR MOLDING
When larger castings are floor molded, nodularity decision is not as time urgent but casting identification and traceability is more important, with cast date and/or heat number cast on the casting.

For example, from a ladle or questionable nodularity, two castings could be poured, one housing and one base. These questionable castings could be in various corners of the foundry surrounded by good castings poured the day before and the day after. Obviously, if final decision is made to reject this questionable ladle, these two castings must be positively identified and removed from the system. While the final decision is being evaluated castings should be tagged and isolated to prevent accidental mixing.

On larger castings, certification reports are frequently required which include record of all analyses, micros, and test data.

HOLDING PROCEDURE AND QUARANTINED AREA
It is important to have clearly established procedures and responsibility for separating and holding low nodularity batches, or any other quality deficiencies like casting defects, dimensions out of tolerance, etc.

Castings in question are usually “red tagged”, noting the reason for holding and held in a quarantined area. Some producers lock the quarantined area to prevent mixup of unreleased castings. Some customer audits require a locked quarantine area.

It is important to have technicians adequately trained to quickly recognize and hold questionable nodularity and casting defects.

It is essential that highest trained quality supervisors or managers make final decisions after retests and close-evaluation. Backup personnel should be designated in case the final decision maker is absent.

A Company Quality Control Manual should include clear instructions on control tests, persons authorized to hold and persons authorized to make final decisions to pass, reject, or reclaim.
DEFICIENCES ENCOUNTERED IN NODULARITY CONTROLS
Consultants have experienced several typical deficiencies in nodularity control. Five more common deficiencies are described.

#1 MICRO CHECK ON EACH LADLE THOUGHT NOT NECESSARY
Several foundries have insisted that a micro was not needed on every ladle because they "did everything the same way, and nothing “was changed”

At one foundry, when a micro on every ladle was insisted upon, on the first day, 24% of the ladles were held for low nodularity or carbides. Carbides were reclaimed by heat treatment on 5% of the ladles, but 19% of production was scrapped due to low nodularity.

In this sandwich treatment operation, where reportedly “no changes were made,” the following twelve variables were observed that caused low magnesium recovery and low nodularity ladles:
- Rusty cover steel. Loose fluffy cover.
- Irregular pour from a too full furnace.
- One shallow ladle used for large casting line.
- Pocket eroded out on one ladle.
- Temperature increased 80F for smaller castings with no magnesium alloy adjustment.
- Higher sulfur on first heat after gray iron.
- Excessive fines in bottom of alloy container.
- New lot or alloy with lower magnesium and lower cerium.
- Slag left, entangling alloy.
- Back iron left causing early ignition of alloy.
- Overweight ladle from over pouring.

Also observed were five post inoculation variables that caused carbides.
- Ferrosilicon inoculant sometimes dumped instead of flowed smoothly into stream. Cup on a handle was available but erratically used.
- Excessive ferrosilicon fines in bottom of container purchased cheaper inoculant sized 1/2 inch X down.
- Half or slag left from treatment ladle, wrapping up ferrosilicon.
- Pouring time too long, normally 6 to 8 minutes was delayed to 16 minutes. Inoculation faded.
- Inoculant amount of .30% silicon was marginal for so many variables. Amount was doubled to adequately cover the many variables.

Adequate control has never been observed where only occasional micros were checked.

#2 INADEQUATE TRAINING AND STANDARDS
In another foundry, a micro was checked on each ladle and "recorded on a nodularity record". Foundry personnel cut the specimens and delivered to an office clerk who polished, examined and recorded.

However, the record made was simply "OK" under nodularity, with no indication of percentage, and no standards on the wall to indicate what percentage was OK.

In checking the micro samples, one was found with about 50%-60% nodularity. It was shown to office clerk-technician who was asked "what level is OK"? She said, "see all those balls". Her OK standard seemed to be a conspicuous volume of nodules or possibly
more nodules than vermicular and flake. She had been trained by the previous office clerk.

This is a graphic example or inadequate training of a micro technician made worse by absence of visual standards and oversimplified reporting. With these inadequacies, training of new technicians became progressively weaker.

**#3 GOOD CONTROLS BUT NO RECORDS**

A small, all ductile foundry, on one shift, had good quality "controls, because the conscientious manager gave personal attention to all critical operations. Microsamples were polished by foundry personnel and placed by the microscope for evaluation by the manager. But no records were made. The manager even stopped sending out tensile tests because he could determine expected properties from the microstructure.

It was forcefully explained to him that records of his good controls were necessary to protect against product liability suits and to pass audits of some customers that his new owners expected to serve.

A good record system was established and orders received from some more demanding purchasers.

**#4 GOOD CONTROL ON FIRST SHIFT BUT GAP ON SECOND SHIFT**

One foundry had excellent controls, trained technicians, and responsibility assignments on a one shift operation. When a skeleton, second shift was added, the spectrometer, micro, and tensile testing technicians were spread out. A micro technician checked nodularity for the first half of the second shift. But, through the last hours of the night shift, micro specimens were poured but saved for an early morning technician to quickly evaluate.

They were warned that this "gap" in their otherwise good system could let some low nodularity castings get by. Before correction could be made, a good customer on a spot check round a low nodularity casting and returned several thousand castings. Each casting was passed through an Ultrasonic Tester. Only 18 low nodularity castings were found, and these were traced to a late hour on the second shift when micros were saved till the next morning. Further tracing revealed that this ladle was the last one treated from a lot of alloy before going to a new lot. It was believed that excessive fines in the bottom of the container caused low magnesium recovery and low nodularity.

This experience emphasizes that if a control system is not completely “air tight” low nodularity castings can get through.

**#5 POOR HOLDING SYSTEM - LOW NODULARITY REPORTED BUT SHIPPED BY MISTAKE**

In one foundry, an inadequate procedure stated only that micro technician should “report any low nodularity ladle to his supervisor”, with no procedure described for isolation, holding, and disposition responsibilities.

A low nodularity ladle was recorded and presumably reported on a Friday afternoon as the casting operation ended for the week. Several days later when the low nodularity report was noticed, it was found that the castings had been processed by night shift cleaning crew.
OTHER GUIDANCE FROM MICROSTRUCTURE
From the quick micro examination, the most immediate and most important objective is verification of acceptable nodularity and absence of carbide.

Other guiding indications can be obtained from the micro. When etched with 2% nital, the amount of pearlite can be determined. Fig. 15 shows four levels of pearlite, 25%, 50%, 75% and all pearlite, no ferrite. When the specimen is a smaller section than the tensile test block and castings represented, pearlite in the specimen will be higher than the tensile bar, but difference can be determined. When the pearlite content of the specimen varies up and down, it can signal that adjustments need to be made for better uniformity.

NODULE COUNT
Nodule count is becoming increasingly useful as an indication of favorable conditions. Fig. 16 shows nodule count standards ranging from 25 to 300 nodules per square millimeter.

DIS Research Project #12 studied “The Influence of Foundry Variables on Nodule Count”.

SECTION THICKNESS through its effect on solidification rate showed the most positive influence, increasing nodule count with decreasing thickness, in the following approximate relationship:

- 1/2 in. section approx 100 nodules per square millimeter
- 1/2 in. section approx 200 nodules per square millimeter
- 1/4 in. section approx 300 nodules per square millimeter
- 1/8 in. section 400-1000 nodules per square millimeter

Other variables affecting nodule count were:

- Increasing post inoculant amount from .25% Si to .75% Si increased nodule count, roughly 150 nodules per square millimeter
- Nodule count decreased with inoculant fade time.
- Sprue and in-mold booster inoculants increased nodule count.
Fig 15. Pearlite proportions in matrix 25% to 100%
Fig 16. Nodule count standards
- In-mold treatment produced some or the higher nodule counts.

With high nodule count, carbides are less likely and most, properties are better.

Preferred nodule count range has been found to be 100 to 200 nodules per square
millimeter in the one inch tensile "Y" block and in the castings.

A well nucleated high nodule count iron also has more smaller grains with less
segregated grain boundary constituents which are unfavorable to best properties,
especially in very heavy sections with long solidification times.

NODULARITY VERIFICATION WITH ULTRASONIC AND RESONANT FREQUENCY
INSTRUMENTS

Individual castings can be tested for nodularity on Resonant Frequency and Ultrasonic
instruments. These tests must be performed on the same position of a casting with the
same thickness and surface condition.

Velocity of the ultrasonic waves is affected by the graphite shape and nodularity. These
instruments must be calibrated to standards established from microsamples.

Some foundries use the ultrasonic instrument to double check nodularity on critical
applications. Others use instruments to sort out mixed lots or marginal conditions within
a ladle of castings.

IN-MOLD castings must be verified by such instruments since each casting is subject to
treatment variables and misses, and no micro sample can be taken to represent a
treatment batch.

Some high production operations, and especially those pouring from a reservoir of
treated metal from a holder, are able to control nodularity without a micro on every ladle.
With frequent spectrometer analyses for Mg and important elements, it is possible to
maintain uniformity and nodularity control with micros taken at established intervals
consistent with process control assurance.

CHAPTER 6

TENSILE TEST VERIFICATION OF GRADE COMPLIANCE

Another important and necessary test is the tensile test to verify that the minimum
properties or the specified grade are being met.

Some special tests sometimes better represent special challenges in service. But, the
tensile test has traditionally been used to indicate general properties of various metals.
From the several grades available, a grade of ductile iron is chosen to provide the
properties desired in the engineering application of the part. The typical grades and
properties specified were described in Chapter 2 and in more detail in Appendix II on
specifications.

For tensile verification, at least one or two tensiles per grade, per shift should be tested
and recorded.
If the quality control system and records indicate that all castings have the same hardness range and matrix structure as the tensile bars, then it can be safely assumed that all the castings within that group meet the tensile specifications.

**Some customers require that tensile bars be cast on to the casting.**

Most tensile bars are cast separately in either "Y" block molds or double leg keel block molds developed by steel foundries.

In order to reduce machining, some foundries cast a rounded double leg keel block with a slotted wafer core on to. A near round bar is cast, fed through the open slot, which requires less machining.

If castings are heat treated, the tensile bar blocks should be heat treated with the castings.

Tensile bars are then machined to dimensions specified by ASTM and pulled according to ASTM procedures.

Machining techniques and surface conditions can affect test results, especially elongation.

For best control, tensile tests should be performed in the foundry within a day for prompt verification, retest, reclamation or rejection.

Foundries that must send tensiles out to commercial laboratories should insist upon prompt reporting within a day or two.

Tensile test results on ductile iron should be reported in a separate book or section rather than being mixed in with tests on gray iron, malleable iron, etc.

Ideally, each grade should be recorded on a separate page so that a scan of results will indicate any "drift" or trends.

If hardness, microstructure and analysis are recorded along with tensile properties, much guidance can be developed toward the amount or pearlite needed and the best alloy combinations to obtain that pearlite level.

SPC graphs or tensile properties facilitate better control.

**CONTROL OF MATRIX IS ESSENTIAL**

After the most critical verification or adequate nodularity and absence of carbides, the next important control is assurance of the matrix required to meet the tensile specification of the selected grade.

In all but the special grades, this matrix control involves obtaining the right proportion or ferrite and pearlite.

One or the attractive features or ductile iron is ability to meet several tensile grades as-cast.

Heat treatment can be used to obtain or correct the matrix needed. Details of various heat treatments are described in Appendix III. Castings with carbides can be reclaimed with a full anneal which includes a high temperature period which decomposes carbides.
Castings with too much pearlite to meet high ductility grades can be slow cool annealed to decompose all pearlite and obtain a high elongation ferritic matrix.

Likewise, castings with insufficient pearlite to meet high strength grades can be air cooled in a normalizing heat treatment. The fast cooling retains pearlite.

Heat treatment for reclamation can be cheaper than scrapping castings. However, if heat treatment is required to meet properties that are usually met as-cast, cost is inflated by the cost of heat treatment and risk of dimensional distortion.

Some customers do not allow heat treatment.

Most foundries try to meet all specifications by controlling the matrix through control or chemistry and cooling rate.

FACTORS INFLUENCING FERRITE AND PEARLITE PROPORTIONS
Ductile Iron Society Research Project #5 showed that tendencies toward ferrite or pearlite first depend on a number of regular variables which determine how much alloying is needed to obtain the pearlite level needed for high strength grades.

Factors decreasing pearlite tendency (from Research Project #5):
- Increased section size, slower cooling
- Increased inoculation. Higher nodule count accompanies
- Lower magnesium residual
- Increased carbon and silicon, and carbon equivalent

A typical step bar in the project illustrates the decline in pearlite as section thickness increased:

<table>
<thead>
<tr>
<th>STEP BAR SECTION THICKNESSES</th>
<th>1/8”</th>
<th>¼”</th>
<th>½”</th>
<th>1”</th>
<th>1 ½”</th>
<th>2”</th>
</tr>
</thead>
<tbody>
<tr>
<td>% Carbide</td>
<td>4%</td>
<td>0%</td>
<td>0%</td>
<td>0%</td>
<td>0%</td>
<td>0%</td>
</tr>
<tr>
<td>% Pearlite</td>
<td>25%</td>
<td>24%</td>
<td>19%</td>
<td>16%</td>
<td>13%</td>
<td>10%</td>
</tr>
</tbody>
</table>

ALLOYING FOR PEARLITE
Since most variables favorable to nodularity favor ferrite formation, the best way to obtain a sufficiently pearlitic matrix is by adding alloys to stabilize pearlite.

The favored alloy is copper which stabilizes pearlite but does not cause carbides. Additions of 0.30% to 0.60% copper will usually produce roughly 50% pearlite to meet 80-55-06 grade. High purity electrolytic copper should be used. Secondary copper can contain contaminants like lead, arsenic, antimony, hydrogen, etc.

Fig. 17 shows increasing proportions of pearlite from left to right with increasing concentrations of copper (on the top) and manganese (on bottom). A fully pearlitic matrix was obtained with 0.82% copper. With 1.47% manganese, the matrix was all pearlite.

Another alloy that forms pearlite and does not form carbide is tin. Tin is very powerful. 10 times as powerful as copper. Tin additions of 0.03%-0.08% can produce a predominantly pearlitic matrix.
Because of its power, the use of tin is not recommended unless very good spectrometer calibration and awareness is available.

Nickel can help meet high strength grades because nickel increases the strength of ferrite and is a mild graphitizer.

The amount of alloy addition required depends on manganese content and other trace alloys like chromium, molybdenum, vanadium, etc. The amount also depends on the other variables previously discussed.

Manganese also assists in the formation of pearlite, but has disadvantages promoting carbides and increasing grain boundary segregation.

It is not recommended that manganese be added. But, less select steel scrap with higher manganese contents or .40-.70% can be economically attractive and used on pearlitic grades.

Some foundries have found it helpful to adjust final alloy additions to total a certain ALLOY FACTOR.

A simple formula used by some is:

\[
\text{ALLOY FACTOR} = \%\text{Cu} + \%\text{Mn} + \%\text{Cr} + \%\text{Mo} + \%\text{Va} + 10 \times \%\text{Tin}
\]

Fig 17. Increased pearlite contents (from left to right) on top from increased copper concentration on bottom from increased manganese

Fig 95. Comparison between the pearlite promoting effects of Mn and Cu, TC 3.85 percent, Si 2.0 percent, etched, original approximately 100X.  
Some find that:

For 80-55-06 grade Alloy Factor 110-130 will meet
For 100-70-03 grades Alloy Factor 140-160 is needed

Some have developed a more complex formula with multipliers for each element.

Each foundry should determine from experience the best formula and total alloy factor for their conditions.

With good records and SPC techniques, the best alloy factor ranges can be determined by experience for various levels of pearlite and strength.

**HIGH STRIPPING TEMPERATURE INCREASES PEARLITE**

If castings are stripped too early and too hot, pearlite will be increased by the faster cooling in air.

The matrix structures expected in ductile iron are based on the assumption that the castings will cool slowly in the sand mold to temperatures below the critical temperature.

Some foundries with short conveyor runs built for malleable iron (or gray iron) have found it difficult to make ferritic grades of ductile iron as-cast because castings are stripped red hot. They find it necessary to anneal in order to meet ferritic high ductility grades.

Many foundries have extended their cooling runs, with conveyor loops stretched out, or loops added upstairs or downstairs in order to obtain slow cooling in the mold, and produce high ductility grades without annealing.

**MATRIX VERIFICATION BY HARDNESS TESTING**

Brinell hardness readings on castings provide an indication of amount of pearlite and strength-ductility combination.

The Brinell is the most useful hardness test on irons because the large impression more accurately includes a total representation of all microconstituents.

If graphite form is nodular and no carbides are present, then Brinell hardness correlates closely with matrix structure. Hardness increases with increased pearlite in the matrix.

On the highest ductility grades, some specifications and customers require a minimum Brinell, like 179, which insures a ferritic matrix with virtually no pearlite. Some customers specify a hardness range.

Hardness correlates with tensile strength when good nodularity and freedom from carbides are assured.

Tensile strength runs 420-450 times Brinell hardness on ferritic grades and 470-480 times on pearlitic grades. This ratio is called K factor. If this K factor ratio of tensile to Brinell is lower it indicates either carbides, poor graphite or unsound specimen.
When hardness is specified, high volume foundries pass castings by a highly automated Brinell machine located in a finishing line for fast Brinell reading and compliance with a specified hardness range.

Some foundries have reduced their variability by using Statistical Process Control techniques and graphs to narrow the hardness range, pearlite variation and property variation in their castings.

MATRIX VERIFICATION WITH EDDY CURRENT INSTRUMENT
An instrument based on Eddy Current, can indicate pearlite level when properly calibrated.

Some foundries use such an instrument in the finishing line for matrix check on all castings for certain customers and applications.

EFFECTS OF SECTION SIZE ON MICROSTRUCTURE
As section size increases, the slow cooling affects microstructure in the following ways:
- Pearlite decreases, ferritic increases
- Nodule count decreases
- Grain size and nodule size increases
- Nodule shape tends to deteriorate, especially if nodulizing conditions are marginal

When castings are produced with sections 3 in. to 6 in., a 3 in. or 4 in. test bar is frequently used for tensile test, to be more representative of the castings.

Some customers require that tensile tests be trepanned from casting sections.

ALL METALS EXPERIENCE DECLINE IN PROPERTIES IN THICKER SECTION
Some reduction in properties is experienced in all metals from the general coarsening or microstructure in slow cooled heavy sections.

Gray iron strength in an 8 in. section drops to one half the strength of a 1 in. section.

Medium carbon steel in an 8 in. section has half the elongation and 6,000 psi lower strength, compared to a 1 in. section.

Aluminum alloys and high strength bronzes lose considerable ductility even in 2 in.

In well controlled ductile iron, decline in properties is no worse and sometimes less than in other metals. However, frequently in very heavy sections of 8 in. to 15 in., properties show a drastic drop especially in elongation. This drop is accompanied by a drastic deterioration of graphite shape to high proportions of irregular, vermicular, and crab like graphite shapes.

RESEARCH ON VERY HEAVY SECTIONS
An early Ductile Iron Research Project #8, was directed toward learning how to maintain optimum properties in very heavy sections. This early research contributed some knowledge but left some unanswered questions. A current research project is pursuing further better understanding of heavy sections.
Demand for nuclear waste casks has increased interest in maintaining highest possible properties. In Germany, ductile iron has been used successfully for containing nuclear waste. With close controls, they have maintained good properties and passed some impressive tests.

In the first project #8, iron with 37 planned variables was poured into a 1 in. keel block, a 4 1/2" cube, and a 9 1/2"" cube.

The 1" keel block solidified in 5 minutes.
The 4 1/2" cube solidified in 20 minutes.
The 9 1/2" cube solidified in 108 minutes.

As shown in table 4, 33% of heats experienced a sharp drop in tensile properties, chiefly elongation, in the 9 1/2"" cube. Elongations ranged as low as 1% to 5%. This drastic drop is the reason for research.

<table>
<thead>
<tr>
<th>Elongation Range</th>
<th>Number</th>
<th>Percent</th>
</tr>
</thead>
<tbody>
<tr>
<td>15% - 21%</td>
<td>14</td>
<td>38%</td>
</tr>
<tr>
<td>10% - 15%</td>
<td>7</td>
<td>19%</td>
</tr>
<tr>
<td>6% - 10%</td>
<td>4</td>
<td>11%</td>
</tr>
<tr>
<td>1% - 5%</td>
<td>12</td>
<td>33%</td>
</tr>
</tbody>
</table>

However, on 38% of the heats, retention of properties was very good with elongations of 14 to 21% retained in the 9 1/2" cube.

Table 5 shows properties of one of the good retention heats “G” and one of the poor retention heats “P”.

Heat “G” had exceptional keel block properties with 25% elongation, 99% nodularity and desirable high nodule count of 213.

From the 4 1/2" cube, good properties were retained. Elongation dropped to 21%, which was still excellent. Nodularity dropped only to 93%. Nodule count decreased to 100, typical of coarsening expected in heavier sections or all metals.

From the 9 1/2 in cube, elongation dropped to 17% which is still excellent for this section. Nodularity percent dropped to 89%, still good.

Heat “P” typifies those with poor retention and drastic decline in properties.

Keel block properties were very good with 20% elongation and 95% nodularity. Retention was still good from the 4 1/2" cube, with 18% elongation and 90" nodularity.

However, in the 9 1/2" cube, elongation dropped sharply to 4%, and tensile strength dropped to 45,080 psi. Nodularity drop to 53% accounts for the low properties. Half the graphite shapes were irregular, vermicular, or crab like shapes, with some flake.

Occasional sharp drops like this have caused concern among ductile iron producers and users.
This poor heat contained .023% titanium which was apparently not enough to damage the 1” and 4 ½” tests but caused graphite deterioration in the 9 ½” section.

Deteriorated graphite shapes showed the most effect on elongation, some effect on tensile strength, and less effect on yield strength. Fig. 18 shows typical forms or deteriorated graphite found in the center of the 9 1/2 in. cubes with drastic decline in elongation.

<table>
<thead>
<tr>
<th></th>
<th>“G” Good Retention</th>
<th>“P” Poor Retention</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td>1” Keel Block</td>
<td>4 ½” Cube</td>
</tr>
<tr>
<td>TS</td>
<td>67,610</td>
<td>63,00</td>
</tr>
<tr>
<td>YS</td>
<td>47,220</td>
<td>44,000</td>
</tr>
<tr>
<td>%E</td>
<td>27%</td>
<td>21%</td>
</tr>
<tr>
<td>% Nodularity</td>
<td>99%</td>
<td>93%</td>
</tr>
<tr>
<td>Nodule Count</td>
<td>213</td>
<td>100</td>
</tr>
</tbody>
</table>
Fig 18. Forms of deteriorated graphite found in 9 ½” cube from DIS research project #8 on heavy section
This project indicated several favorable and unfavorable variables.

- Pearlite is detrimental to elongation in heavy sections. High manganese and high alloy residuals that retained pearlite gave some of the lowest elongations.
- Post inoculation fading showed strong effect. A ladle held 5 minutes dropped to fair 12.5% elongation. One held 31 minutes dropped seriously to 5.5% elongation.
- With ferrosilicon booster inoculation, in the mold (even after 30 minutes holding) all retained good properties.
- Mold booster inoculation methods included granular ferrosilicon, ferrosilicon tablets, and solid inserts anchored in the mold.
- Several of the highest tests were from heats with booster inoculation in the mold. When good inoculation was maintained, more nodular graphite shapes were retained and higher nodule counts resulted, along with finer grains and less grain boundary segregates.
- Special inoculant compositions containing zirconium, barium, and strontium showed no significant advantage over, standard inoculant grade ferrosilicon within the limits of this experiment. An inoculant with titanium caused the very low 4% elongation in the 9 1/2 in. cube.
- With significant amounts of deleterious trace elements, cerium in the right amount improved properties. With no significant concentrations of trace elements, better properties may be retained without cerium.
- The balance of cerium with trace elements appeared to have some importance, but was not sufficiently developed. (Later experience has developed some formulas for evaluating total residual elements and need for cerium.)
- The major cause of elongation loss was deteriorated graphite shape. Other detrimental constituents are pearlite and segregated grain boundary constituents made worse by manganese, along with molybdenum and vanadium residuals. All these elements segregate to grain boundaries.
- Maintaining more late nuclei from good inoculation encourages the precipitation of finer more nodular graphite as well as finer grains, with less grain boundary segregates.

Use of metal chills in the mold has been found helpful toward shortening the long solidification times and reducing the risk of structural deterioration.

From recent experiences, some heavy section foundries have developed formulas for evaluating the detrimental effect or the total percentages of deleterious elements and the need for cerium.

One such formula for cumulative detrimental effect is:

\[ \text{SG} = 4.4\text{Ti} + 2\text{As} + 2.3\text{Sn} + 5\text{Sb} + 290\text{ Pb} + 370\text{Bi} + 1.6\text{AL} \]

If SG totals less than 1.0, spheroidal graphite is possible. If SG totals more than 1.0, deteriorated graphite is likely and cerium is needed to counteract.

In order to total a safe SG less than 1.0, these residual elements must all be less than half the individual maximum safe limits we have proposed. Lead and bismuth especially must not both be near maximum limits.
Some have determined how much cerium is desirable to neutralize the total residual elements. Much is yet to be learned in this area.

CHAPTER 7

STATISTICAL PROCESS CONTROL - A TOOL FOR BETTER CONTROL

Some of the best progress toward improved quality control has been accomplished by the use of Statistical Process Control (SPC) techniques.

When analyses and tests are graphed, the control picture is readily apparent to workers, supervisors, and management. (7) Workers can take pride in a good quality control "scoreboard". Reasons for variations can be determined and analyzed.

Fullest understanding and utilization of SPC techniques require considerable training. Some large companies have several supervisors with extensive training in SPC techniques to direct their program, and considerable training is provided to the line supervisors and key workers in the processes.

It is beyond the purpose of this guide to give a complete course in all SPC techniques. Hopefully, a briefer indication of usefulness along with some examples of useful, simple graphs that can be utilized by the smallest foundry will encourage development of a SPC program that fits the size of the foundry and the markets served.

CONTROL CHARTS MOST USEFUL

One of the most useful tools is a simple control chart. In ductile iron, control of iron analysis is very important. Control charts of carbon, silicon, sulfur and magnesium contents are very useful guides, as well as any other elements significant to product control.

From pages of written analyses, it is hard to recognize trends. But, from a graph a trend of uniformity or variability is readily apparent.

Fig. 19 (from Booth of Ford Motor) (8) shows a control chart of carbon analyses plotted within upper and lower control limits.

In a small foundry, analyses and tests can be manually plotted by a technician, in the laboratory or on the melting floor, and made visible to all who contribute to the process and its control.

Where a computer is available, the control graphs can be charted by the computer with printouts visible at several stations.
In a few of the most sophisticated systems, a computer controlled announcement is made if one of the elements is out of control or has shown a trend on several successive samples toward out-of-control limits.

Control charts or treatment temperature and pouring temperature are useful as well as some treatment variables like pouring time.

Graphs of tensile properties and hardness readings make variations and trends more evident.

Sand control can be improved by graphs of important sand tests.
Control charts or casting dimensions can lead to less variability and closer dimensional control.

**HISTOGRAM CHARTS DISPLAY PATTERN OF VARIATION**

On some property, like hardness, marks can be made for each reading at various levels, as shown in Fig. 20.

A typically good variation pattern is in the shape of a bell with the most readings in the middle tapering down to a few readings at the low and high extremes.

A narrow, high bell indicates good control within a narrow range of variation. A short, wide bell indicates a wider pattern of variation. A distorted or off center bell indicates more problems and variation toward one of the limits.

Fig. 20 can typify a situation where specified Brinell hardness is 156-217. However, process spread is 180-230. Some percentage of the castings, run higher than the 217 specification maximum BHN requiring heat treatment to reclaim.

The process spread is not greater than the specification spread, but is off center. Further SPC techniques could reflect the cause and bring the bell pattern toward the center of the specification range.

The process capability is adequate to meet present hardness specification if relocated. Further study could lead to improved process capability to meet narrowed ranges with less variability.

**CORRELATION GRAPHS (or SCATTER GRAPHS) SHOW CAUSES**

Graphing an effect against a possible cause can indicate a positive or negative cause relationship.

In the previous example of the hardness, histogram, if hardness was plotted against carbon, sulfur, or magnesium, a "shot gun" scatter would indicate no correlation. However, if hardness was plotted against percent of pearlite, data points would fall close to a line indicating a positive correlation and cause or the hardness variation. Then, if pearlite were plotted against manganese content, some correlation would be reflected. If plotted against alloy combination factor, better correlation would result. Improvement in alloy combination control could bring the bell toward the center of the range, improve process capability and reduce variability.

**PARETO CHARTS graphically portray the major causes or a problem.** For example, when causes of scrap loss are presented as a bar graph proportional to magnitude, then from the many causes of scrap, the "major few" causes can be identified from the "trivial many". Then improvement efforts can be concentrated on the few defects that account for 80% of the loss.

**CORRECTIVE ACTION IS ESSENTIAL**

The advantage of graphical data is fast recognition of the variation pattern and tests approaching or outside control limits. But, a vital part of Statistical Process Control is the corrective controlling response to data out or limits. No benefit is realized until corrective response is made by someone.
Process instructions should describe corrections to be made for every test or analysis out of control limits.

It is important that the person be designated who is responsible for control adjustments on iron analysis, temperature, sand control, and all areas or control.

The company Quality Control Manual should designate persons with corrective control responsibility and decisions in all areas contributing to total casting quality.

Customer audits look for SPC systems as indications of a well controlled operation and good organization for quality emphasis and reliability.

Statistical Process Control is an essential element of TOTAL PROCESS CONTROL.

CHAPTER 8

CONTROLLING CASTING DEFECTS

Previous sections have concentrated on controls toward iron control and metallurgical integrity which is so important to the reputation of ductile iron. However, total casting quality includes freedom from casting defects such as gas porosity, dross, and shrinkage.

A few guiding principles from the Ductile Iron Society Research will be summarized.

GAS POROSITY - "PIN HOLES"
The Ductile Iron Society Research Project #6 investigated eight mold variables and eight metal variables that might affect gas porosity.

A BCIra Pinhole test casting was used which had six wafers ranging in thickness from 3/32" to 3/4" around a central down sprue. Each heat was split, pouring half as gray iron, then half was magnesium treated to ductile iron to determine pin hole tendencies in ductile iron compared to gray iron from the same melt and same type molds.

Types or pin holes experienced and recognized were:
  - Hydrogen pin holes
  - Nitrogen pin holes
  - Carbon monoxide pin holes

Combinations of these gases can contribute once triggered by the primary source.

Fig. 21 shows pin holes considered moderate, and severe.

Ductile iron showed a greater propensity for pin holes than gray iron, showing nearly twice the severity and frequency of gas holes over an average of all experimental variables.
Mold materials showed the following order of frequency and severity of gas holes:

Most pin holes -
- High moisture green sand with low sea coal
- Normal green sand
- High nitrogen no bake
- High nitrogen shell
- Low nitrogen shell
- Nitrogen free no-bake
- Sodium silicate sand

Least pin holes -
- Dry sand

In green sand, increased sea coal content progressively reduced pin holes, eliminating completely at the 4%-5% level. This is attributed to a reducing atmosphere counteracting the oxidizing potential of moisture.

Recycled sand produced more gas holes than new sand.

Section sizes showing most gas hole tendency were ¼” to 1”. In sections under ¼”, rapid solidification prevents gas pickup and bubble growth. In sections above 1”, slower solidification permits diffusion and dissipation of any high concentration of dissolved gas.

Normal nitrogen content as melted was 80 ppm, increased artificially with cyanide to 130 ppm. The turbulence of magnesium treatment flushed nitrogen to near normal levels. But, after treatment, magnesium increases propensity for nitrogen absorption from chemical binders in the mold.

The greatest threat of nitrogen pin holes appears to be high nitrogen chemical binders. Nitrogen content can build up in recycled return.

Normal hydrogen content, as melted, was 2 ppm, increased artificially with mud balls to 4 ppm. Excessive hydrogen is also flushed by magnesium treatment. But, exposure to moisture after treatment produces serious pin holes. The presence or magnesium increases absorption tendency from insufficiently heated troughs, green ladles or wet patches and from high moisture, low sea coal green sand.
Carbon variations and silicon variations showed no significant effect on gas porosity.

Tellurium additions or .002% in ductile iron can eliminate hydrogen pin holes, but tellurium is deleterious to graphite nodularity and surface, and is not a safe medicine. Titanium additions can eliminate nitrogen pin holes in gray iron but titanium is detrimental to graphite nodularity.

Aluminum present at certain levels during melting can increase pin holes from hydrogen absorption. But, aluminum additions after melting have eliminated pin holes by lowering iron oxide content.

Carbon monoxide type gas holes can be formed by reaction of the carbon in the iron with excessive iron oxide from turbulence or mold moisture.

The total gas available to form gas holes appears to be a combination of:

- gas contained in the metal plus
- gas absorbed from mold binders and moisture
- oxidation from turbulence in pouring plus aspiration in the gating system
- particles of entrapped dross and slag can nucleate gas evolving reactions
- formation of a porosity cavity depends upon entrapment of evolving gas during a critical stage of solidification which depends upon mechanics and rate of solidification

There is much yet to be learned about gas porosity. But, freedom from this defect can be accomplished through close control of melting, knowledge or sand binders, close sand control, and diligent ladle practice, temperature control and good gating practice.

DROSS DEFECTS

The casting of all metals involves some risk of oxidation products, inclusions and slag that can be generated and entrapped on casting surfaces, causing defects.

In ductile iron, the tendency toward films and surface inclusions seems to be more complex. Magnesium is reactive to oxygen, and sulfur forming oxide and sulfide inclusions which combine with any sand to form fluid silicate inclusions.

Fig. 22 shows surface defects from magnesium silicate dross sometimes called "elephant skin defect".

Ductile Iron Research Project #11 studied the effect on dross defects of:

- Four metal variables
- Nine mold variables
- Six gating variables

Test specimen was a 4X4X2 1/2 in. block with a 1/4 in. plate attached. A side riser fed the casting.

Dross ratings 1 to 5 were made visually and from magnaflux indications.
Metal variables showed some effects:
- High pouring temperature produced less dross
- High base sulfur showed some more dross
- High magnesium content of .06% and .08% increased dross tendency considerably
- Carbon equivalent variations showed no significant effect

Mold variables showed considerable effect indicating reactivity with the metal:
Worst dross  - High moisture green sand, no sea coal
- No-bake, poor practice
- Dried sand
- No-bake, good practice
- High sea coal green sand
- Sodium silicate sand

Least dross  - Graphite mold

Gating variations showed the most effect. A pressurized system with 4:8:3 ratio produced the least dross. A 1:4:4 ratio was much worse and a 20% choke at the down sprue gave the poorest dross ratings from the same metal and mold materials.

The best gating was choked at the ingate. In-gates branched from the bottom of a high large volume runner that retarded metal velocity and trapped dross and slag in the top or the high runner.
From this research, the following dross explanation was developed:

1. MUCH PRIMARY DROSS reaction products are generated from magnesium treatment. In the turbulence, the coarsest of the magnesium oxides, sulfides and silicates float out and are skimmed off. Then, in post inoculation, some silicates are formed, to be skimmed off. The volume of primary dross is increased by high base sulfur, high magnesium content, oxidation sources, dirty ladles. etc.

Most of this primary dross can be removed with thorough skimming or held back by teapot ladles, skimmer blocks, bottom pouring. etc.

2. SECONDARY DROSS, is generated as the iron containing magnesium first encounters any splashing or turbulence in the gating system and then as the iron runs over the mold. High moisture in green sand is oxidizing but high sea coal is reducing and, neutralizing. Low pouring temperature increases drossy silicates.

3. ENTRAPMENT AND LOCATION ARE DETERMINED BY GATING
A good gating system first minimizes turbulence, and then entraps most or the dross and slag in the runner, with clean metal off the bottom going into the casting cavity.

SLAG REMOVAL
In ductile iron, much slag is generated in the treatment process as magnesium oxides, sulfides and silicates. This slag must be thoroughly skimmed off. Then, post inoculation may produce some slag which should be removed.

SLAG CONDITIONERS
Two types of slag conditioners have proven helpful - SLAG FLUIDIZERS and SLAG COAGULANTS.

SLAG FLUIDIZERS like cryolite, sodium fluoride or calcium fluoride in small additions or 0.002% - 0.10% in the treatment ladle fluidize the reaction inclusions and facilitate their coalescence and floating out with a cleansing effect on the iron.

SLAG COAGULANTS added on top of the slag "ball up" the slag and assist in its removal.

LADLE PRACTICE IMPORTANT TO CLEAN IRON
Treatment and pouring ladles tend to build up slag which becomes increasingly higher in oxides and sulfides. With excessive use this slag as it becomes more fluid will wash off some into the metal. And the high iron oxide content will oxidize some magnesium and generate inclusions and slag and higher sulfur content will re-contaminate the iron.

These dirty ladles should be replaced with new ladles at proper intervals. But, new linings in fresh ladles, if not well preheated, can contribute oxidizing influence and hydrogen from combined water in clay binders.

Ladle patches are seldom heated enough and contribute "wet" moisture and combined water with serious contamination of the iron.

A sufficient number of ladles should be lined and preheated to be readily rotated into service when ladles in service have excessive slag build up.
High alumina refractories are less reactive and preferred for ladle linings. Silica and clay refractories are more reactive magnesium.

**INSULATING LADLE BOARD LININGS**
Cleaner ladle practice has been experienced with the use of insulating board linings in ladles and pouring boxes.

Insulating refractory boards are cut and fitted into a ladle with a castable backup lining and loose sand poured into the space between.

Advantages reported are:
- simpler lining installation and easy removal
- less slag buildup
- no need for preheating
- less temperature loss

**SHRINKAGE POROSITY**
Research project #10 studied factors involved in shrinkage cavities in ductile iron. Fig. 23 sketches three types of shrinkage: "Sink", "Internal Shrinkage Cavity" and "Draw".

Because of its more abrupt solidification, ductile iron experiences more shrinkage and more mold deformation than gray iron.

To determine the effect of volume to surface area ratio, the test specimen chosen was a 12X12 in. plate 3/8 in. thick with cylindrical bosses, ranging from 1 in. diameter to 2 1/2 in. diameter in each corner. A 4 1/2 in. center boss was expected to have considerable shrinkage which was measured by a special water displacement device.

Mold types were green sand and alkyd resin no-bake. Iron variable included carbon equivalent, post inoculation, pouring temperature.

- The greatest factor contributing to shrinkage was confirmed to be mold deformation and enlargement. The most shrinkage occurred with the greatest mold dilation from measurement of boss diameter.

- Green sand molds experienced more deformation and more shrinkage volume than the no-bake molds. Type of mold was a most significant factor.
Volume to surface area ratio showed a definite effect on shrinkage. The 1 in. boss was sound all conditions never experiencing any shrinkage. The ratio of volume to surface area, or modulus was .13. The 2 in. bosses were sound about half the time under the more favorable conditions. The 4 1/2 in. central boss with a modulus of .75 had considerable shrinkage under all conditions. Shrinkage volume measured from 8 ml on the highest carbon equivalent in no-bake mold, to 55 ml in green sand molds with lowest carbon equivalent.

Carbon equivalent showed a significant effect. For shrinkage effect the best carbon equivalent formula, established in the literature, is CEq=C+1/7 Si. In green sand molds, shrinkage volume at the lowest 3.5% CEq was 55 ml or 5%. Shrinkage decreased as CEq increased down to 25 ml or 2.3% at 4.2% CEq. Considerable risering would be necessary for complete soundness in the 2 1/2 in. and 4 1/2 in. cylinders.
- In the no-bake molds, the measured shrinkage decreased from 45 ml or 3.8% at 3.5 CE to 8 ml or 0.6% as CEq increased to 4.2. Some risering would be necessary to be completely free of shrinkage.

- In some very rigid molds, like dry sand and cement molds, ductile iron castings can be made sound without risering. But, in most mold systems, risering or thick sections is necessary for soundness. The extent of risering depends most upon the mold deformation of the mold system and the metallurgical quality of the iron.

**GATING CONTROL IMPORTANT TO CASTING CLEANLINESS**

As previously indicated in the dross research summary, gating variations had the most effect on dross and slag in the casting.

In early years, Karsay with QIT Fer at Titane, Inc., along with others, published some guiding principles and formulas for best gating. The QIT organization has maintained constructive on gating and risering. (9) In Ductile Iron Training Courses, QIT personnel teach the gating and risering section of the course and have published useful guides to gating and risering.

It is not the purpose of this Q. A. Guide to give a complete course on gating and risering. It is the intention only to emphasize the importance of certain principles and provide some guidance toward an organized system.

Several gating systems have been used with some degree of success with certain types of castings.

The system described here has been most successful with least dross and inclusions in the most difficult circumstances.

Ductile iron should be poured as fast as possible but with minimum splashing and turbulence.

In the recommended pressurized gating system, the choke or smallest area (that determines pouring rate) should be the in-gate into the casting, or total area or multiple in-gates.

Fig. 24 provides a graph from which recommended pouring time is indicated for various casting weights. For example, a 100 lb. casting should be poured in 9 seconds. Tables and graphs are also available to quickly provide gate area for recommended pouring rate.

Fig. 25 graphically provides the choke cross section area of all in-gates for the recommended pouring time for castings of various weights. For example, the 100 lb. casting, if in the cope, should have .65 sq. in. or in-gate area. If the casting is in the drag, 0.55 sq.in. of in-gate area.

Several gating alignment principles are recommended as shown in Fig. 26:

- A well at the bottom or the down sprue prevents splashing and avoids turbulence at the direction change.
- A runner extension beyond the furthest in-gate traps the dross and slag usually concentrated on the leading edge of the stream.

- A high runner with height at least twice the width tends to collect much of the dross and slag in the top or the runner.

- A runner cross section area greater than in-gate area and sprue area tends to retard the velocity of the stream and encourage "floating out" of dross and slag in the runner.

- In-gates should be shallow and wide branching flush from the bottom of the runner, providing the cleanest metal from the bottom of the runner into the casting cavity.

The gating ratio that has the best record for clean castings is 4:8:3, which represents the ratio of cross section areas of down sprue, to runner, to in-gate. Some have experienced good success with a 4:6:3 ratio which incorporates the important relationships but with slightly smaller runner area.

Fig 24.

Fig 25.
Fig 26. Alignment of horizontal parting system

Fig 27. Gating arrangements for vertically parting molds

Fast but smooth continuous pouring is important, without interruption and without turbulence. The runner system should be filled as soon as possible and kept full.
For fullest effectiveness of this system, downsprues of various diameters must be available in order to maintain this ratio.

When gate area is first chosen to provide the recommended pouring time" then a down sprue with 30% to 50% more area must be chosen in order to keep the runner system full and choked, with a pressurized system maintained.

Then, runner area 2 to 2 ½ times total in-gate area is designed with height at least 2X width.

Gating of vertically parted molds involve the same principles but some different techniques. Fig. 27 shows 7 gating arrangements. Systems I, II and III filling mold cavities from the bottom tend to provide cleaner castings but with lower yield. Systems VI and VII filling cavities from the top provide the best yield but freedom from slag defects depends entirely on slag free iron down the sprue.

Responsibility for gating and risering should be assigned to one person with training and experience.

If records are kept of gating dimensions and alignment principles and correlated with loss records and defect occurrence, gating performance can be continually improved.

POURIN BASINS on top of the down sprue help maintain smooth continuous pouring, break the impact of the pouring stream, and can hold back any ladle slag that enter the stream. Basins can be justified on medium and large castings but seldom on small castings where the ladle lip can be positioned close to the mold sprue, and the pouring stream can be controlled more smoothly.

CERAMIC FILTERS properly placed in the gating system can improve cleanliness of the iron into the mold by entrapping inclusions, sand, and slag.

Filters should not be used as a "crutch" substitute for a good gating system, but should be used to enhance a good gating system.

Filters are especially helpful with in-mold treatment and are good insurance on complex castings with much intricate molding and coring.

The recommendations or filter manufacturers should be followed in choice of size and porosity area in order to maintain proper flow rates in the gating system.

**RISERING FOR SOUND CASTINGS**

As pointed out in the shrinkage research, thick sections in most types or molds experience a net shrinkage of 2% to 5% which will leave shrinkage porosity in the center of thicker sections if not fed with riser feed metal.

Steel castings have more shrinkage and steel foundrymen have developed from research and experience many principles of riser types, and sizes, and feeding distance necessary to soundly feed various sections.

Gray iron experiences less net shrinkage and requires less risering. During graphitization, some expansion counteracts some of the overall shrinkage.
Ductile iron has a solidification behavior different from steel and gray iron.

Ductile iron does not solidify in progressive layers like steel but has a more instantaneous eutectic type solidification.

The graphitization process provides some expansion which counteracts some of the liquid contraction and solidification shrinkage. In very rigid molds this graphitization expansion makes it possible to produce sound castings without risers.

But, in most mold types, the instantaneous solidification and graphite expansion increase mold deformation more in ductile iron. And, mold deformation increases the need for risering.

Professor Heine has contributed useful papers on shrinkage, solidification patterns, and risering. (17).

QIT personnel have published “Essentials of Gating and Risering Design” and teach these subjects in D.I.S. Training Courses (9).

It is beyond the scope of this guide to present a complete course on risering. It is intended only to provide some guiding principles and to emphasize the need for risering responsibility to be assigned to one person with training and experience.

Castings not too thick and with a progressive solidification opportunity, are sometimes fed by the gating system without the need for risers.

Thick castings can be made without risers in rigid molds like dry sand or cement molds. With no swelling of the mold, the expansion during the graphitization stage leaves no net shrinkage and no need for a riser.

One type of specialized risering is PRESSURE CONTROL RISERING which utilizes the expansion phase then freezes off the riser neck exactly at the time when freezing and expansion of the casting begins. This system may permit a more efficient yield, but depends on thorough understanding of a limited set of circumstances.

CONVENTIONAL RISERING is more generally adaptable and better understood. Its success depends on directing the freezing from cold points through heavier sections, and placing a riser of suitable size at the end of each freezing path, attached to the last section to solidify. The heavier section (with highest modulus) feeds the thinner sections, then draws feed metal from the riser.

The riser should be cylindrical in shape with diameter greater than the thickest section being fed. Height above the highest point of the casting determines feed volume available. Heine has described how risers can be tapered toward the top. (17)

Blind risers are preferable to open top risers but must have a dimple or pencil core in the top to keep open to atmospheric pressure.

Riser contact or neck can be reduced some to facilitate knock off. But, if the area of the neck is less than about 60% of the riser area or too much less than the casting thickness, the riser connection may freeze off too early before all shrinkage has been fed. Short necks and sharp notches permit more reduction in area.
Exothermic sleeves are available in various diameters with built-in necks. The heat generated from the exothermic material enables a smaller diameter riser to feed heavy sections.

Fig. 28 shows gating into a riser with notched riser connection into the casting.

![Fig 28. Gating into riser with notched connection to the riser](image)

Fig 29. Three types of blind risers

![Fig 29. Three types of blind risers](image)

Fig 30. Typical side riser showing connection, height and volume

![Fig 30. Typical side riser showing connection, height and volume](image)
Fig. 29 illustrates three types of blind risers, type I, a side riser with contact in the cope, type 2, a side riser with contact in the drag, and type 3, a top riser.

Fig. 30 shows a typical side riser, design of connection with a notch, and effective riser height and volume.

Fig. 31 is a useful graph provided by QIT that readily provides feed metal volume for risers of various diameters and effective heights.

A major decision in risering is where risers are needed and the best location for effectiveness.

The first step is evaluation of cooling rates of various casting sections.

The modulus of a section, which is volume/surface area, is the best indication of cooling rate. A thin 1/4 in. plate has a low modulus of .12 and a fast cooling rate. While a thick 2 in. plate has a modulus of 1.0 and a slower cooling rate.

A cylinder has a higher modulus than a rectangle of the same volume.

The casting is first divided into segments. The shape of each segment is determined, whether more nearly a cube, plate, rectangular bar, a round bar, or a cylinder, etc.

Then, the modulus of each segment is calculated. If there is a progression from thinnest low modulus section to the thickest high modulus section, then gating into one riser attached to the thickest section will feed the entire casting.

A more complex case is where a section in the middle is thinner (lower modulus) than sections on each end. In this case, the modulus of the center section is considered a transfer modulus as illustrated in Fig. 32. In order for pressure control risering to be effective, expanding liquid must be able to refill a riser used to feed liquid shrinkage. This ability to feed metal is determined by calculating the transfer modulus and assessing the metallurgical quality.

If modulus of sections 2 and 4 are high enough, and the metallurgical quality is high, the single riser on the end will feed all sections. However, if the modulus of section 2 is too low, it will freeze off before it can feed section 1, and the single riser will not feed section 1. Another riser will be needed on the other end.
Fig 31. Graph providing riser feed metal volume (QIT)

Fig 32. Evaluating modulus of transfer section

Metal chills strategically located in the mold can accelerate solidification of heavier sections, improve the progressive pattern of solidification, simplify risering and improve metal structure.

Computerized solidification graphics have made possible the modeling of the progression of solidification, indicating the first and last areas to freeze and simplifying the choice and location of risers.
Solidification graphics can also point the way to design improvements that will either facilitate production or improve service performance.

Metallurgical quality of ductile iron is a factor that will influence amount or shrinkage and feeding requirement.

Iron that has been well inoculated and has high nodule count has less net shrinkage than an iron with low nodule count with coarse grain and nodules, and likelihood of some carbides.

Pouring temperature influences shrinkage and should be controlled within a range best suited for the risering system.

Inconsistent sand control and mold rammed hardness can cause variations in mold deformation and shrinkage volume.

Castings which generally are sound with no shrinkage can occasionally encounter shrinkage either if metallurgical quality of the iron declines increasing the shrinkage of the iron or if sand goes out of control in a direction to increase mold deformation and casting volume to be fed.

Consistent soundness requires consistent controls of all elements of the process.

SURFACE FINISH - PENETRATION
Casting finish is sometimes unsatisfactory due to a surface defect called penetration. George DiSylvestro has commendably summarized several types and causes of penetration. (11) Fig. 33 shows sketches of the two most common types "burn on" and "burn in" penetration.

The "burn on" type penetrates no further than the depth of one or two sand grains, and can usually be blasted off, but leaves a rough surface. "Burn in" type penetrates more than the depth of two grains of sand and surface finish is very rough and unacceptable.
Fig. 7. Schematic sketch of burn-on penetration Type 1A.

Fig. 8. Schematic sketch of burn-in penetration Type 1B.

Fig 33. Penetration types – “Burn on” or “burn in”

Fig 34. Chemical reaction type of penetration
Possible causes are:
- oxidizing mold conditions
- insufficient refractoriness of sand
- inadequate ramming compaction
- excessive pouring temperature

Remedies are:
- add new sand
- increase the volatile content
- increase mold compaction
- control pouring temperature
- use a mold wash effectively

Other more specialized types of penetration are described in the sand literature such as:
- Hard mold penetration
- Chemical reaction penetration
- Vapor state penetration
- Water explosion penetration
- Mold explosion penetration
- Eutectic exudation

Fig. 34 shows a casting with chemical reaction penetration. Iron has been oxidized due to excessive turbulence or moisture and the oxide has reacted with the molding sand.

When excessive surface roughness is encountered, reasons can be found in sand test reports.

SAND CONTROL IMPORTANT TO CASTING QUALITY
Previous sections have indicated how poorly controlled sand can contribute to penetration, gas holes, and dross, as well as excessive mold deformation which increases shrinkage to be fed.

Adequate sand testing and sand control are necessary for total quality of castings produced.

Basic process control tests needed at intervals are:
- Moisture
- Compactability
- Density
- Green Compression
- Permeability

More specialized tests needed daily are:
- Combustibles
- Methylene blue test for active clay
- Dry strength
- Sieve test
- Temperature of return sand

Useful mold tests are:
- Hardness
- Mold strength
Some useful visual observations indicating sand condition are:
- Britteness
- Amount of steam
- Ease of draw
- Breakdown in cooling line and shake-out
- Appearance of uncleaned castings and blasted castings

Statistical Process Control graphs and techniques greatly improve sand control.

Targets and limits are clearly displayed. The sand technician, by marking test values on a control chart, can readily recognize trends and out of control conditions.

Sand control charts can portray the total picture of sand condition for the assurance and guidance of sand preparation personnel, molding supervision, and overall quality managers.

CASTING SKIN CONDITIONS*

FERRITIC SKIN:
When producing pearlitic ductile iron sometimes a thin surface layer of ferrite is observed. This may be due to marginal magnesium levels in the iron. Magnesium besides being nodulizer is also a strong pearlite promoter. If the magnesium level in the surface of the casting is depleted due to oxidation during mold filling then ferritic skin may be observed. In this case magnesium residual is adequate to form nodular graphite but not enough to promote pearlite.

To minimize the ferritic skin effect one or more of the following steps can be taken.

1) Magnesium residuals must be adequate.
2) Cerium containing inoculants may be used.
3) Reduce oxidation of metal by improving the gating system.
4) Reduce the oxidizing atmosphere in the mold. (Reducing moisture, increasing carbonaceous material)
*Contributed by Al Alagarsamy, Director R&D, Grede Foundries

PEARLITIC RIM AND FERRITIC SKIN:
In some rare cases a very thin layer of pearlite on the surface followed by ferritic layer is observed. This also for much the same reasons previously mentioned except the graphite is no longer nodular in the pearlitic layer. Flake graphite is present in the surface which promotes pearlite formation. In addition to magnesium depletion there may be metal mold reaction resulting in oxygen and sulfur diffusion into the casting surface layer forming flake graphite. Adjacent to this flake graphite layer graphite is nodular but the matrix is ferrite due to marginal magnesium as discussed in the previous paragraph.

In addition to the steps mentioned before to minimize ferritic skin, sulfur levels in the sand should be monitored and kept low to prevent flake graphite formation in the surface.

DIMENSIONAL CONTROL
An essential control in the production of all castings is control of dimensions within the tolerances required for service performance. It is important that critical dimensions, and dimensional tolerances be agreed upon with the customer before the start of production.
The casting producer must make many dimensional allowances for processing steps.

Various metals require different pattern makers allowance for shrinkage.

Mold deformation increases external dimensions depending on type of sand binder, size of casting etc.

Most heat treatments cause small dimensional changes which must be anticipated.

It is general practice for the first trial castings from a new pattern or for a new application, to be given a complete dimensional check using calipers and layout tables. Dimensional checks are a part of final inspection.

Complex instruments, like Coordinate Measuring Machines, Laser Measuring, Replication Microscope, etc. make possible faster and more effective checks of dimensions.

FINAL INSPECTION
A final inspection for casting defects is a vital element of quality reliability.

Visual inspection can be aided with pictures describing possible defects and locations to be expected with indications of unacceptable magnitude or dimensions. Inspectors need to be thoroughly trained.

Critical dimensions may require gaging.

If hardness range is specified, in-line hardness testing is necessary.

Anyone of several non destructive tests may be required to insure against sub-surface defects.

Magnaflux can detect defects just below the surface with orientation of magnetic powder.

Radiography or X-ray can detect any unsoundness at any depth.

Ultrasonic testing is sometimes used to verify nodularity of each casting. Ultrasonic tests can also be used to detect internal unsoundness.

Eddy current testing of each casting is sometimes used to verify the amount of pearlite in the matrix.

On all non-destructive tests, standards should be clearly understood and agreed upon with the customer before production is started, and cost of special tests should be recognized.
CHAPTER 9
QUALITY ORGANIZATION-AUDIT EXPECTATIONS

RESPONSIBILITIES ESTABLISHED
In order to supervise all the controls and tests involved in total casting quality, it is necessary to have an overall Quality Control Manager responsible for all tests and records. The Quality Control Manager should preferably have a Technical Degree or several short courses in metallurgy and quality control.

Several well trained technicians are needed to make the tests on the various processes. In the absence of the Quality Control Manager, an alternate must be designated to make the decisions which require prompt action, such as nodularity.

Technicians should be trained to hold castings or call attention to elements out of control. Some supervisor, in every area, must be trained and authorized to make prompt corrections of tests out of control limits.

The size of the Quality Control staff increases with the size of the company and the volume and complexity of production.

PROCESS FLOW CHART
A process chart can provide graphic guidance to production personnel and confidence to the purchaser. Example 1 shows one such flow diagram indicating the steps in casting production and the controls along the way.

ANALYSIS CONTROL RANGE
A form containing all analysis specifications and expectations is also helpful to production control and customer confidence. Example 2 shows a typical form for analysis expectations, frequency, and responsibility for control.

COMPANY QUALITY CONTROL MANUAL
A company Quality Assurance Manual or Process Control Manual should establish management commitment to quality, outline controls, tests and limits for each process, and responsibilities for controls and quality decisions. A system for casting identification and traceability must be provided and described.

Records of all tests should be kept in a manner that can be related to identifiable castings. Nodularity verification of each batch must be described.

Holding procedures should be outlined for low nodularity or any tests out of specification. Holding in a quarantine area is recommended with a "hold tag" indicating reason for holding.

Sufficient tensile tests should be recorded to verify meeting tensile specifications of the grade. Procedures and responsibilities should be described for calibration of test equipment, Spectrometer, etc. Description of Statistical Process Controls used within the quality system should be outlined. Responsibility should be assigned for control decisions within each process and for final decisions on acceptable quality by the Quality Control Manager.
Example 1.
Example 2.

<table>
<thead>
<tr>
<th>NO.</th>
<th>CHARACTERISTIC</th>
<th>LIMITS</th>
<th>HOW OFTEN</th>
<th>HOW TO MEASURE</th>
<th>WHO</th>
</tr>
</thead>
<tbody>
<tr>
<td>01</td>
<td>Correct Carbon Content</td>
<td>0.26% ± 0.08%</td>
<td>Each Heat</td>
<td>Spectrometer/Leco</td>
<td>Mett Labo</td>
</tr>
<tr>
<td>02</td>
<td>Correct Silicon Content</td>
<td>0.50± ± 0.10%</td>
<td>Each Heat</td>
<td>Spectrometer/Leco</td>
<td>Mett Labo</td>
</tr>
<tr>
<td>03</td>
<td>Correct Carbon Equivalent</td>
<td>See Below</td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td></td>
<td>A. Less than 0.5&quot;(12mm) section</td>
<td>4.45 to 4.53%</td>
<td>Each Heat</td>
<td>Analysis</td>
<td>Mett Labo</td>
</tr>
<tr>
<td></td>
<td>B. 0.5-2.0&quot;(12-50mm) section</td>
<td>4.40 to 4.50%</td>
<td>Each Heat</td>
<td>Analysis</td>
<td>Mett Labo</td>
</tr>
<tr>
<td></td>
<td>C. Over 2.0&quot;(50mm) section</td>
<td>4.30 to 4.40%</td>
<td>Each Heat</td>
<td>Analysis</td>
<td>Mett Labo</td>
</tr>
<tr>
<td>04</td>
<td>Correct Manganese Content</td>
<td>0.03% ± 0.05%</td>
<td>Each Heat</td>
<td>Spectrometer/Leco</td>
<td>Mett Labo</td>
</tr>
<tr>
<td>05</td>
<td>Correct Sulphur Content</td>
<td>0.02% ± 0.005%</td>
<td>Each Heat</td>
<td>Spectrometer/Leco</td>
<td>Mett Labo</td>
</tr>
<tr>
<td>06</td>
<td>Correct Phosphorus Content</td>
<td>See Below</td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td></td>
<td>A. Copper Ferronisi</td>
<td>0.03% maximum</td>
<td>Each Heat</td>
<td>Spectrometer/Leco</td>
<td>Mett Labo</td>
</tr>
<tr>
<td></td>
<td>B. High Temperature Irons</td>
<td>0.10% maximum</td>
<td>Each Heat</td>
<td>Spectrometer/Leco</td>
<td>Mett Labo</td>
</tr>
<tr>
<td>07</td>
<td>Correct Magnesium Content</td>
<td>0.03% ± 0.01%</td>
<td>Each Heat</td>
<td>Spectrometer/Leco</td>
<td>Mett Labo</td>
</tr>
<tr>
<td>08</td>
<td>Correct Cerium Content</td>
<td>0.003% ± 0.003%</td>
<td>Each Heat</td>
<td>Spectrometer/Leco</td>
<td>Mett Labo</td>
</tr>
<tr>
<td>09</td>
<td>Correct Aluminium Content</td>
<td>See Below</td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td></td>
<td>A. 0.03-0.045% Mg Iron</td>
<td>0.04% maximum</td>
<td>Each Heat</td>
<td>Spectrometer/Leco</td>
<td>Mett Labo</td>
</tr>
<tr>
<td></td>
<td>B. 0.045-0.055% Mg Iron</td>
<td>0.03% maximum</td>
<td>Each Heat</td>
<td>Spectrometer/Leco</td>
<td>Mett Labo</td>
</tr>
<tr>
<td>10</td>
<td>Correct Chromium Content</td>
<td>See Below</td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td></td>
<td>A. Ferritic Irons</td>
<td>0.05% maximum</td>
<td>Each Heat</td>
<td>Spectrometer/Leco</td>
<td>Mett Labo</td>
</tr>
<tr>
<td></td>
<td>B. Pearlitic Irons</td>
<td>0.10% maximum</td>
<td>Each Heat</td>
<td>Spectrometer/Leco</td>
<td>Mett Labo</td>
</tr>
<tr>
<td>11</td>
<td>Correct Nickel Content (if req)</td>
<td>0.05% ± 0.10%</td>
<td>Each Heat</td>
<td>Spectrometer/Leco</td>
<td>Mett Labo</td>
</tr>
<tr>
<td>12</td>
<td>Correct Moly Content (if req)</td>
<td>See Below</td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td></td>
<td>A. Ferritic Iron</td>
<td>0.03% maximum</td>
<td>Each Heat</td>
<td>Spectrometer/Leco</td>
<td>Mett Labo</td>
</tr>
<tr>
<td></td>
<td>B. Pearlitic Iron (if req)</td>
<td>0.05% ± 0.05%</td>
<td>Each Heat</td>
<td>Spectrometer/Leco</td>
<td>Mett Labo</td>
</tr>
<tr>
<td></td>
<td>C. High Temp. Irons (if req)</td>
<td>0.10% ± 0.10%</td>
<td>Each Heat</td>
<td>Spectrometer/Leco</td>
<td>Mett Labo</td>
</tr>
<tr>
<td>13</td>
<td>Correct Vanadium Content</td>
<td>0.02% maximum</td>
<td>Daily or if required</td>
<td>Spectrometer/Leco</td>
<td>Mett Labo</td>
</tr>
<tr>
<td>14</td>
<td>Correct Antimony Content</td>
<td>0.003% maximum</td>
<td>Daily or if required</td>
<td>Spectrometer/Leco</td>
<td>Mett Labo</td>
</tr>
<tr>
<td>15</td>
<td>Correct Arsenic Content</td>
<td>0.01% maximum</td>
<td>Daily or if required</td>
<td>Spectrometer/Leco</td>
<td>Mett Labo</td>
</tr>
<tr>
<td>16</td>
<td>Correct Bismuth Content</td>
<td>0.002% maximum</td>
<td>Daily or if required</td>
<td>Spectrometer/Leco</td>
<td>Mett Labo</td>
</tr>
<tr>
<td>17</td>
<td>Correct Boron Content</td>
<td>0.003% maximum</td>
<td>Daily or if required</td>
<td>Spectrometer/Leco</td>
<td>Mett Labo</td>
</tr>
<tr>
<td>18</td>
<td>Correct Copper Content</td>
<td>See Below</td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td></td>
<td>A. Ferritic Ductile</td>
<td>0.05% maximum</td>
<td>Each Heat</td>
<td>Spectrometer/Leco</td>
<td>Mett Labo</td>
</tr>
<tr>
<td></td>
<td>B. Pearlitic Ductile (as req)</td>
<td>0.05% ± 0.10%</td>
<td>Each Heat</td>
<td>Spectrometer/Leco</td>
<td>Mett Labo</td>
</tr>
<tr>
<td>19</td>
<td>Correct Lead Content</td>
<td>0.002% maximum</td>
<td>Each Heat</td>
<td>Spectrometer/Leco</td>
<td>Mett Labo</td>
</tr>
<tr>
<td>20</td>
<td>Correct Tellurium Content</td>
<td>0.02% maximum</td>
<td>Weekly or if required</td>
<td>Spectrometer/Leco</td>
<td>Mett Labo</td>
</tr>
<tr>
<td>21</td>
<td>Correct Tin Content</td>
<td>See Below</td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td></td>
<td>A. Ferritic Irons</td>
<td>0.02% maximum</td>
<td>Daily or if required</td>
<td>Spectrometer/Leco</td>
<td>Mett Labo</td>
</tr>
<tr>
<td></td>
<td>B. Pearlitic Irons</td>
<td>0.10% maximum</td>
<td>Daily or if required</td>
<td>Spectrometer/Leco</td>
<td>Mett Labo</td>
</tr>
<tr>
<td></td>
<td>C. High Temperature Irons</td>
<td>0.10% maximum</td>
<td>Daily or if required</td>
<td>Spectrometer/Leco</td>
<td>Mett Labo</td>
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<tr>
<td>22</td>
<td>Correct Titanium Content</td>
<td>See Below</td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td></td>
<td>A. Up to 2&quot;(50mm) sections</td>
<td>0.07% maximum</td>
<td>Each Heat</td>
<td>Spectrometer/Leco</td>
<td>Mett Labo</td>
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<tr>
<td></td>
<td>B. Over 2&quot;(50mm) sections</td>
<td>0.05% maximum</td>
<td>Each Heat</td>
<td>Spectrometer/Leco</td>
<td>Mett Labo</td>
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<tr>
<td>23</td>
<td>Correct Selenium Content</td>
<td>0.03% maximum</td>
<td>Weekly or if required</td>
<td>Spectrometer/Leco</td>
<td>Mett Labo</td>
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</table>
CUSTOMER AUDITING EXPECTATIONS

Increasingly, more purchasers or castings are requiring an audit of the quality organization and procedures of the prospective casting supplier.

Purchaser representatives visit and inspect records, tests, and analyses and personnel qualifications of those enforcing quality and making final decisions.

Customer audit requirements of several of the most demanding purchasers have been reviewed to develop the list below.

Following are typical items looked for in the most demanding customer audits:

ORGANIZATIONAL ITEMS LOOKED FOR:
- Management commitment to quality
- Quality Control Manual or Process Control Manual
- Qualified Manager of Quality with authority
- Sufficiently trained Quality Control personnel and technicians.
- Written instructions for each operation
- Clearly defined responsibilities
- Use of Statistical Process Control Techniques
- On-going training programs
- Casting identification and traceability system
- Rework and salvage procedures, authorization responsibility
- Non conformance holding procedures, tags
- Quarantine area (some require caged and locked)
- Evidences of employee involvement, team approach
- Attitudes conducive to quality improvement and

TOTAL ORGANIZATION COMMITMENT

PROCESS CONTROL ITEMS TYPICALLY LOOKED FOR ARE:

- Purchased materials specifications
- Quality evaluation of suppliers
- Raw materials analyses, inspection
- Process flow charts and control plans
- Melting controls
- Analysis ranges - C, Si, P, S, Mg, Mn, residual elements
- Treatment controls
- Temperature controls, records
- Nodularity verification by micro or instrument. Records of nodularity
- If heat treated, temperature cycles
- Tensile tests, records
- Microstructure checks and records
- Brinell hardness as required
- Use of SPC control charts and techniques
- Sand control tests and control limits
- Molding control points. Core making controls
- Dimensional checks, tolerances, measurement methods and frequency
- Check of patterns and core boxes for wear
- Visual inspection for defects
- Calibration procedures, and schedule for test equipment, gages, Spectrometer, etc.
- Adequacy of inspection stations and instructions
- Special requirements for "safety parts"
- Records maintained for five years

FUTURE QUALITY IMPROVEMENTS

The general level of metallurgical quality of ductile iron has progressively improved over recent years. Longtime volume producers have determined that their quality index, based on tensile properties, today is higher than 20 years ago.

Further improvements and reduced variability can be expected in future years.

Casting soundness, surface finish, and dimensional control expectations are being raised.

Research is continuing to show the way toward improved properties and casting integrity.

As ductile iron and austempered ductile iron go into more challenging applications, it can be expected that special tests like fatigue strength, impact and fracture toughness will become more important.

As these advances take place, revisions in this Quality Assurance Guide will be needed.

The main focus has been on concise straightforward controls and actions required for the quality expectations of today’s market.

Several specialized subjects and more detailed explanations are included in ten appendices that follow.
APPENDIX I

COMPLETED RESEARCH PROJECTS OF THE DUCTILE IRON SOCIETY
(distributed to members)

1. COOPERATIVE DEVELOPMENT OF SPECTROMETER STANDARDS FOR DUCTILE IRON FOR NBS 1967
2. REVISION OF IRON-CARBON-SILICON PHASE SYSTEM (High C Area)
   Prof. Heine and Olen at University of Wisconsin, January 1968
3. DISSOLUTION, DESULFURIZATION AND PRESSURE RELATIONSHIP OF MAGNESIUM VAPOR IN DUCTILE IRON
   Profs Speer and Parlee at Stanford University, September 1971
4. FACTORS INFLUENCING OCCURRENCE OF CARBIDES IN DUCTILE IRON
   Prof. Wallace and Dubrava at Case Western University, January 1972
5. INFLUENCE OF PROCESSING VARIABLES ON MATRIX STRUCTURE AND NODULARITY OF DUCTILE IRON
   Prof. Wallace and Dubrava at Case Western University, September 1972
6. FACTORS INFLUENCING PIN HOLES IN GRAY AND DUCTILE IRON
   Prof. Wallace, Evans and Harkness at Case Western University, April 1974
7. TIME TEMPERATURE RELATIONSHIPS IN DECOMPOSITION OF CARBIDES
   Prof. Engel and Datta at Georgia Institute of Technology, November 1975
8. FACTORS AFFECTING OPTIMUM PROPERTIES IN HEAVY SECTION DUCTILE IRON
   Prof. Wallace and Helmink at Case Western University, September 1977
9. EFFECT OF TRACE ELEMENTS AND COMPOSITION VARIABLES ON ANNEALING TIME OF DUCTILE IRON
   Prof. Wright and Glover at University of Alabama, June 1979
10. FACTORS INFLUENCING SHRINKAGE IN DUCTILE IRON
    Prof. Wallace and Sarnal at Case Western University, October 1981
11. FACTORS INFLUENCING DROSS FORMATION IN DUCTILE IRON
    Prof. Wallace and Samal at Case Western University, December 1982
12. INFLUENCE OF FOUNDRY VARIABLES ON NODULE COUNT
    Profs Wallace, Du and Su at Case Western University, April 1984
13. INFLUENCE OF ALLOY COMPOSITION AND TRACE ELEMENTS ON NODULE COUNT
    Profs. Stefanescu, Chen and Martinez at University of Alabama, April 1984
14. FACTORS INFLUENCING MACHINABILITY OF DUCTILE IRON
    Prof. Berry et al at Georgia Institute or Technology, October 1987
15. FRACTURE TOUGHNESS AND IMPACT EVALUATION OF DUCTILE IRON
    Prof. Bradley et al at Texas A & M, January 1989
16. CHARACTERIZATION OF INCLUSIONS IN DUCTILE IRON
    Richard Gundlach, August 1989

RESEARCH PROJECTS IN PROCESS IN 1992

IMPACT PROPERTIES OF DUCTILE IRON BY DYNAMIC TEAR TESTING

MACHINABILITY OF DUCTILE IRON (further study)

MAINTAINING BEST PROPERTIES IN HEAVY SECTION DUCTILE IRON

DETERMINATION OF DYNAMIC TEAR VALUES THROUGH ACOUSTICAL MEASUREMENT
APPENDIX II

SPECIFICATIONS FOR DUCTILE IRON

In the early years, after the discovery of ductile iron, specifications were established in several countries by various specification organizations. Table 6 summarizes ductile iron specifications by American organizations.

ASTM A536 is a popular general specification with five grades representing different strength-ductility combinations.

The highest ductility grade is 60-40-16. To meet the 16% elongation, most producers anneal, but some meet as-cast with close controls.

The 65-45-12 is a popular ferritic grade usually met as-cast. The 80-55-06 is a popular pearlitic high strength grade met as-cast with some alloy additions.

S.A.E. specifications specify hardness ranges for the same 5 grades. Hardness is a good reflection of strength and ductility, if nodularity is assured and carbides are avoided.

MIL-1-22243 requires annealing to meet the highest elongation of 20% with 55,000 psi strength and 37,000 psi yield. This specification requires an impact of 10 ft. lbs. on a charpy V-notch at 20F.

AWWA C151 for pressure pipes requires 60-42-10 from tensile specimen from the wall of the pipe, and an impact of 7 ft. lbs. on a modified charpy V specimen utilizing the full pipe wall.

French specifications NFA-32-201 have grades in metric denominations which translated roughly to lbs per. sq. in. are:

<table>
<thead>
<tr>
<th>TENSILE 1000 psi</th>
<th>YIELD 1000 psi</th>
<th>% ELONGATION</th>
</tr>
</thead>
<tbody>
<tr>
<td>54</td>
<td>34</td>
<td>15</td>
</tr>
<tr>
<td>60</td>
<td>40</td>
<td>12</td>
</tr>
<tr>
<td>71</td>
<td>50</td>
<td>7</td>
</tr>
<tr>
<td>85</td>
<td>57</td>
<td>2</td>
</tr>
<tr>
<td>100</td>
<td>67</td>
<td>2</td>
</tr>
</tbody>
</table>
Table 6. Summary of Ductile Iron Specifications

<table>
<thead>
<tr>
<th>Specification Number</th>
<th>Class or Grade</th>
<th>Min Tensile Strength (MPa)</th>
<th>Min Yield Strength (MPa)</th>
<th>Elongation in 2 in. (%)</th>
<th>Heat Treatment</th>
<th>Other Requirements</th>
</tr>
</thead>
<tbody>
<tr>
<td>ASTM A536-77</td>
<td>80-40-18</td>
<td>60,000</td>
<td>40,000</td>
<td>18</td>
<td>May be annealed</td>
<td>Chemical composition is subordinate to mechanical properties; however, the content of any chemical element may be specified by mutual agreement, hardness Bhn (c) 120 max, microstructure (d) ferritic.</td>
</tr>
<tr>
<td>SAE J434b</td>
<td>85-45-12</td>
<td>80,000</td>
<td>55,000</td>
<td>12</td>
<td>May be annealed</td>
<td></td>
</tr>
<tr>
<td>80-55-08</td>
<td>80,000</td>
<td>55,000</td>
<td>5</td>
<td></td>
<td>Usually normalized</td>
<td></td>
</tr>
<tr>
<td>100-70-03</td>
<td>100,000</td>
<td>70,000</td>
<td>3</td>
<td></td>
<td>Quenched and tempered</td>
<td></td>
</tr>
<tr>
<td>120-90-02</td>
<td>120,000</td>
<td>90,000</td>
<td>2</td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>11468A (Ordinance)</td>
<td>D-4018</td>
<td>60,000</td>
<td>40,000</td>
<td>18</td>
<td>May be annealed</td>
<td></td>
</tr>
<tr>
<td>0-4512</td>
<td>65,000</td>
<td>45,000</td>
<td>12</td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>D-5506</td>
<td>80,000</td>
<td>55,000</td>
<td>5</td>
<td></td>
<td>May be normalized</td>
<td></td>
</tr>
<tr>
<td>D-7003</td>
<td>100,000</td>
<td>70,000</td>
<td>3</td>
<td></td>
<td>Quenched and tempered</td>
<td></td>
</tr>
<tr>
<td>D38T</td>
<td>By agreement</td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>11468A (Ordinance)</td>
<td>D-4018</td>
<td>60,000</td>
<td>40,000</td>
<td>18</td>
<td>May be annealed</td>
<td></td>
</tr>
<tr>
<td>0-4512</td>
<td>65,000</td>
<td>45,000</td>
<td>12</td>
<td></td>
<td></td>
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<td>D-5506</td>
<td>80,000</td>
<td>55,000</td>
<td>5</td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>D-7003</td>
<td>100,000</td>
<td>70,000</td>
<td>3</td>
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<td></td>
<td></td>
</tr>
<tr>
<td>11468A (Ordinance)</td>
<td>60,000</td>
<td>60,000</td>
<td>3</td>
<td></td>
<td>Normalized</td>
<td></td>
</tr>
<tr>
<td>60-40-03(g)</td>
<td>60,000(g)</td>
<td>40,000</td>
<td>18</td>
<td></td>
<td>May be annealed, Bhn 143-187</td>
<td></td>
</tr>
<tr>
<td>11468A (Ordinance)</td>
<td>ASTM A39577</td>
<td>80,60-03(g)</td>
<td>60,000</td>
<td>3</td>
<td>To be used in as-cast condition, hardness shall be minimum Bhn 201</td>
<td></td>
</tr>
<tr>
<td>ASME SA295</td>
<td>60,000</td>
<td>60,000</td>
<td>3</td>
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<td></td>
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<tr>
<td>MIL-I-24137</td>
<td>Class A</td>
<td>60,000</td>
<td>45,000</td>
<td>15</td>
<td>Shall be ferritised by annealing to Bhn 190 max (h)</td>
<td></td>
</tr>
<tr>
<td>(Ship)</td>
<td>MIL-22243</td>
<td>55,000</td>
<td>37,000</td>
<td>20</td>
<td>Ferritised, see details in specifications</td>
<td></td>
</tr>
<tr>
<td>amended</td>
<td>10 ft-lb min</td>
<td></td>
<td></td>
<td></td>
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<td></td>
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<tr>
<td>Charpy V-notch at 20°F</td>
<td>2.20</td>
<td></td>
<td></td>
<td>Max</td>
<td></td>
<td></td>
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<tr>
<td>AMS 5316</td>
<td>60,000</td>
<td>45,000</td>
<td>15</td>
<td>Announced</td>
<td></td>
<td></td>
</tr>
<tr>
<td>60-40-18(k)</td>
<td>60,000</td>
<td>40,000</td>
<td>18</td>
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<tr>
<td>ASTM A445-71</td>
<td>AMS 531B</td>
<td>60,000</td>
<td>45,000</td>
<td>15</td>
<td>Announced</td>
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<td>API 604</td>
<td>60,000</td>
<td>45,000</td>
<td>15</td>
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<tr>
<td>AMS 5315</td>
<td>60,000</td>
<td>60,000</td>
<td>3(i)</td>
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<tr>
<td>AGMA 244.02</td>
<td>65,000</td>
<td>45,000</td>
<td>10</td>
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</tr>
<tr>
<td>AGMA 244.02</td>
<td>70,000</td>
<td>55,000</td>
<td>7</td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>AGMA 244.02</td>
<td>85,000</td>
<td>70,000</td>
<td>5</td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>AGMA 244.02</td>
<td>85,000</td>
<td>75,000</td>
<td>4</td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>AGMA 244.02</td>
<td>100,000</td>
<td>87,000</td>
<td>3</td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>AGMA 244.02</td>
<td>107,000</td>
<td>92,000</td>
<td>2</td>
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<td></td>
<td></td>
</tr>
<tr>
<td>AGMA 244.02</td>
<td>115,000</td>
<td>100,000</td>
<td>1.5</td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>AGMA 244.02</td>
<td>120,000</td>
<td>105,000</td>
<td>1</td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>AGMA 244.02</td>
<td>123,000</td>
<td>123,000</td>
<td>0.5</td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>AGMA 244.02</td>
<td>123,000</td>
<td>123,000</td>
<td>0.5</td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>AGMA 244.02</td>
<td>130,000</td>
<td>82,000</td>
<td>8</td>
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</tr>
<tr>
<td>AGMA 244.02</td>
<td>110,000</td>
<td>90,000</td>
<td>4</td>
<td></td>
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</tr>
<tr>
<td>AGMA 244.02</td>
<td>106,000</td>
<td>105,000</td>
<td>5</td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>AGMA 244.02</td>
<td>120,000</td>
<td>105,000</td>
<td>5</td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>AGMA 244.02</td>
<td>125,000</td>
<td>110,000</td>
<td>2.5</td>
<td></td>
<td></td>
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<tr>
<td>AGMA 244.02</td>
<td>158,000</td>
<td>130,000</td>
<td>1</td>
<td></td>
<td></td>
<td></td>
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<tr>
<td>AGMA 244.02</td>
<td>60,000</td>
<td>42,000</td>
<td>10</td>
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<td></td>
<td></td>
</tr>
<tr>
<td>AWWA C151</td>
<td>Pressure Pipe</td>
<td>60,000</td>
<td>42,000</td>
<td>10</td>
<td></td>
<td></td>
</tr>
</tbody>
</table>

<table>
<thead>
<tr>
<th>Composition, %</th>
<th>TC</th>
<th>Si</th>
<th>P</th>
<th>Other</th>
<th>CE (1)</th>
<th>Bhn</th>
</tr>
</thead>
<tbody>
<tr>
<td>Min Max</td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>60-40-18</td>
<td>3.0</td>
<td>2.5</td>
<td>0.08</td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>60-40-03(g)</td>
<td>3.0</td>
<td>3.0</td>
<td>0.08</td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>60-60-03(g)</td>
<td>3.0</td>
<td>3.0</td>
<td>0.08</td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>60-40-18(k)</td>
<td>3.0</td>
<td>2.5</td>
<td>0.08</td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>60-40-18(k)</td>
<td>3.0</td>
<td>2.5</td>
<td>0.08</td>
<td></td>
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<td></td>
</tr>
</tbody>
</table>

82
German specifications DIN 1693 also have 5 grades which translated to psi are:

<table>
<thead>
<tr>
<th>TENSILE 1000 psi</th>
<th>YIELD 1000 psi</th>
<th>% ELONGATION</th>
</tr>
</thead>
<tbody>
<tr>
<td>57</td>
<td>36</td>
<td>15</td>
</tr>
<tr>
<td>71</td>
<td>46</td>
<td>7</td>
</tr>
<tr>
<td>85</td>
<td>54</td>
<td>3</td>
</tr>
<tr>
<td>100</td>
<td>63</td>
<td>2</td>
</tr>
<tr>
<td>114</td>
<td>71</td>
<td>2</td>
</tr>
</tbody>
</table>

Two German specifications specify impact properties.

The British have 12 grades in UK standard B52789 as shown in Table 7, along with some useful data on expected properties beside tensile properties.

The three ferritic grades are:
- 51-31-22
- 58-38-18
- 61-42-12

Their three pearlitic grades and three hardened and tempered grades are similar except for yield strength.

With increase in international trade, the variations among specifications of various countries have encouraged the establishment of international standards by the International Standards Organization (I.S.O.).

The Table 8 lists six grades specified by I.S.O. 1083 with hardness ranges and matrix structure. These are:

- 116-70-02
- 102-61-02
- 87-54-03
- 73-46-07
- 58-36-12
- 54-33-17

The highest ductility grade has an impact specification of 9.5 rt. lbs (or 13 Joules).

Specifications specify minimum test values, and some were established in the earlier years of ductile iron experience.
Table 7. Some mechanical properties expected in ductile iron grades covered by UK standard B278

<table>
<thead>
<tr>
<th>Grade</th>
<th>Tensile strength</th>
<th>Yield strength</th>
<th>Elongation (%)</th>
<th>Yield strength in compression</th>
<th>Shear strength</th>
<th>Torsional strength</th>
<th>Modulus of elasticity (GPa)</th>
<th>Modulus of rigidity (GPa)</th>
<th>Poisson’s ratio, ν</th>
<th>Hardness, HB</th>
<th>Fatigue limits</th>
</tr>
</thead>
<tbody>
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<table>
<thead>
<tr>
<th>Grade</th>
<th>Tensile strength</th>
<th>Yield strength</th>
<th>Elongation (%)</th>
<th>Yield strength in compression</th>
<th>Shear strength</th>
<th>Torsional strength</th>
<th>Modulus of elasticity (GPa)</th>
<th>Modulus of rigidity (GPa)</th>
<th>Poisson’s ratio, ν</th>
<th>Hardness, HB</th>
<th>Fatigue limits</th>
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</table>

Intermediate grades

<table>
<thead>
<tr>
<th>Grade</th>
<th>Tensile strength</th>
<th>Yield strength</th>
<th>Elongation (%)</th>
<th>Yield strength in compression</th>
<th>Shear strength</th>
<th>Torsional strength</th>
<th>Modulus of elasticity (GPa)</th>
<th>Modulus of rigidity (GPa)</th>
<th>Poisson’s ratio, ν</th>
<th>Hardness, HB</th>
<th>Fatigue limits</th>
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</tbody>
</table>

Pearlitic re-cast and normalized

<table>
<thead>
<tr>
<th>Grade</th>
<th>Tensile strength</th>
<th>Yield strength</th>
<th>Elongation (%)</th>
<th>Yield strength in compression</th>
<th>Shear strength</th>
<th>Torsional strength</th>
<th>Modulus of elasticity (GPa)</th>
<th>Modulus of rigidity (GPa)</th>
<th>Poisson’s ratio, ν</th>
<th>Hardness, HB</th>
<th>Fatigue limits</th>
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</tbody>
</table>

Hardened-and-tempered grades

<table>
<thead>
<tr>
<th>Grade</th>
<th>Tensile strength</th>
<th>Yield strength</th>
<th>Elongation (%)</th>
<th>Yield strength in compression</th>
<th>Shear strength</th>
<th>Torsional strength</th>
<th>Modulus of elasticity (GPa)</th>
<th>Modulus of rigidity (GPa)</th>
<th>Poisson’s ratio, ν</th>
<th>Hardness, HB</th>
<th>Fatigue limits</th>
</tr>
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<tbody>
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</tbody>
</table>

Table 8. Ductile iron property requirements of International Standards Organization

<table>
<thead>
<tr>
<th>Grade</th>
<th>Tensile strength</th>
<th>Yield strength</th>
<th>0.2% offset yield strength</th>
<th>Elongation</th>
<th>Impact energy</th>
</tr>
</thead>
<tbody>
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<td></td>
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</tbody>
</table>

ISO Standard 1083 (International)

<table>
<thead>
<tr>
<th>Grade</th>
<th>Tensile strength</th>
<th>Yield strength</th>
<th>0.2% offset yield strength</th>
<th>Elongation</th>
<th>Impact energy</th>
</tr>
</thead>
<tbody>
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<td></td>
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</table>

High quality producers with good controls generally exceed the minimum specified properties and have no trouble meeting specifications of any of the countries or I.S.O. specifications.

When both the tensile specifications of ASTM and the hardness specifications of SAE are imposed, some conflict can result as pointed out in a paper by Janowak and Alagarsamy (18).

On graphs of Brinell hardness against tensile properties, a scatterband was shown, resulting from chemistry and process variables plus measurement errors.

When hardness was on the low end of SAE specifications, occasional tensile tests would not meet the ASTM requirements for tensile or sometimes yield strength. And when hardness readings were near the maximum of SAE specifications, some tests failed to meet ASTM specification for elongation.

For control guidance, the general relationship between hardness and tensile properties can be helpful. But, for specification acceptability either SAE hardness or ASTM tensile properties should be dominant, and the other property considered as 'typical' or 'generally expected'.

It is important that specifications be understood and agreed upon between the casting producer and buyer before production starts.

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APPENDIX III

HEAT TREATMENTS FOR DUCTILE IRON
Ductile iron is a family of engineering materials with various combinations of properties obtained by matrix control. One attractive advantage of ductile iron is that at least three distinctive grades can be produced as-cast. Several more grades can be obtained by heat treatment. Any of the conventional heat treatment methods used for ferrous materials can be applied to ductile iron.

Grades normally produced as-cast, but missed, can be corrected by heat treatment. Ferritic grades with carbides and/or too much pearlite can be heat treated to a ferritic structure by annealing.

Pearlitic high strength grades with insufficient pearlite can be corrected by an air cool normalizing heat treatment.

Hot shakeout causes more pearlite and sometimes necessitates annealing for ferritic grades.

FULL ANNEAL
A hypercritical full anneal first decomposes any carbides at a temperature of 1650-1750°F for one to two hours per inch of section. Lower temperature requires more time.

Then, pearlite is broken down in the range of 1200-1400°F, which can be a gradual cooling from the high carbide temperature with 2 to 3 hours in the 1400-1200°F range.

Faster pearlite breakdown can be accomplished with an intermediate cooling below 1200°F and heating back to 1300-1400°F range.

TYPES OF HEAT TREATING FURNACES
Annealing furnaces can be car type batch furnaces in which the high temperature is obtained and then dropped to lower temperature to complete the cycle. Or, annealing furnaces can be continuous furnaces with castings pushed through various controlled temperature zones at a speed to provide the necessary time at temperature.

Pressure pipes are annealed in long horizontal furnaces 120 to 200 ft. long. Pipes are pushed through a 1700°F carbide breakdown zone for approximately 1 to 2 hours, cooled below 1200°F, then through a 1400 to 1200°F pearlite breakdown zone for approximately 2-3 hours. This full anneal develops a ferritic, carbide free, matrix with optimum ductility and toughness.

SUBCRITICAL ANNEAL
If carbides are known to be absent, pearlite can be broken down to ferrite with a simpler lower temperature of 1250-1300°F for five hours plus one hour per inch of section, cooling slowly to 1100°F then air cooling.

A subcritical anneal provides the best impact properties, with the highest impact energy and the lowest transition temperature.
EFFECT OF COMPOSITION AND TRACE ALLOYS ON ANNEALING TIME
Recognizing that time for carbide breakdown and pearlite breakdown are affected by certain alloys and composition variables, Ductile Iron Society Research Project #9 was designed to determine effects of these major variables on annealing time.

Specimens were from metal mold with .20-30% carbides
- from thin resin bonded molds with 5% carbides
- from sand cast step bars with no carbides for study of pearlite annealing only.

Annealing time for both carbide and pearlite were shortened by high carbon, and high nodule count from good inoculation. High silicon contents near and above 3.00% greatly shortened annealing time but are unfavorable to impact properties. Increased section thicknesses and high Mg contents required more time.

Annealing temperature greatly influenced the speed of breakdown of both carbides and pearlite. Breakdown of the 30% carbides in metal molds required
- at 1600F 105 minutes
- at 1700F 90 minutes
- at 1800F 45 minutes

But, the higher temperatures increase the risk of warpage.

Breakdown of pearlite to less than 10% required
- at 1300F 90 minutes
- at 1350F 75 minutes
- at 1420F 60 minutes

But, the 1420F temperature is dangerously close to the pearlite reformation temperature for some compositions and is no~ safe.

Chromium content of .17% doubled carbide annealing time and increased pearlite annealing time 50%.

With chromium of .31%, it required over 6 hours to reduce pearlite to 10%, and was essentially impractical to break down all pearlite.

Copper at .31% caused no increase in carbide breakdown time but increased pearlite annealing time 50%.

With tin at .03% level, it required over 5 hours to reduce pearlite to 10% and appeared to require an impractical time to anneal to no pearlite.

This confirmed why pearlitic returns with copper or tin additions should not be charged into ferritic heats.

Trace levels of arsenic, boron, antimony, and bismuth increased pearlite breakdown time 50% and more.

NORMALIZING HEAT TREATMENT
Normalizing is a treatment to produce pearlite by air cooling from 1600-1700F. Castings should be held at this austenitizing temperature for two hours per inch of section then cooled in air. Castings with sections greater than 1 inch may require fan cooling.
Step normalizing produces a structure not as high in pearlite with lower strength and higher ductility. After austenitizing at 1650-1700°F for 2 to 5 hrs, castings are furnace cooled to 1375 to 1450°F, held for 3 hours, and then air cooled.

Tempering after normalizing will improve toughness and impact resistance. Tempering involves reheating to a tempering temperature of 800°F to 1200°F and holding for 2 hours per inch. Higher tempering temperature yields lower hardness and higher ductility.

QUENCHING AND TEMPERING
As in steel, the highest strength grades of ductile iron can be obtained by a quench and temper heat treatment.

The property levels of the 120-90-02 grade are obtained by quenching and tempering.

Castings are austenitized at 1550-1650°F for 2-3 hours, then quenched into oil which is circulated. This fast quench develops a hard brittle martensite which should be tempered as soon as possible to a tempering temperature of 800 to 1200°F. Temper temperature is chosen for the strength and hardness desired. Tempering time should be 2 hours plus 1 hour per inch wall thickness.

Fig. 35 shows a quench and tempered microstructure of tempered martensite.

Thick sections are difficult to quench and encounter some risk of cracking. Alloying may be advisable to improve hardenability.

AUSTEMPERING which is a specialized quench into a salt bath at controlled temperature is described in Appendix V as a specialized family of ductile irons.

STRESS RELIEVING
Stress relieving is not intended to change the structure but to relieve stresses in order to minimize cracking or distortion after machining.

Recommended temperatures for stress relieving are: 950 to 1100°F for one hour per inch of section for unalloyed compositions, and 1100-1250°F for alloyed compositions, followed by furnace cooling to 550°F, then air cooling.

SURFACE HARDENING
Like steel, ductile iron can be surface hardened by several methods to develop a hard wear resistant case while retaining a core with good ductility and impact resistance.

FLAME HARDENING heats the surface with torch flames to 1550-1600°F, then quenches, usually in water. Since the surface is heated only to a shallow depth, hardening occurs only to that depth. Surface hardness ranges from Rockwell C 40 to 60 depending on the core structure.

INDUCTION HARDENING involves the same heat cycle but uses electric induction to heat the surface before quenching. Case depths of .02 to .04 inches are usually developed.

NITRIDING in cracked ammonia for 2 to 3 hours can develop surface hardness of 60 Rockwell C.

HEAT TREATING CONTROLS AND RECORDS
Obtaining the desirable structures by heat treatment requires close control of furnace temperature with thermocouples properly located to reflect casting temperatures. Most furnaces have automatic control of temperature that can be set. And, time and temperature are printed on charts. Heat treating charts should be a part of the quality control files.

When castings are heat treated, tensile test bars should be placed in the middle of the casting load, and given the same heat treatment as the castings.

![Tempered martensite in structure of quenched and tempered ductile iron](image)

Fig. 35 Temper martensite in structure of quenched and tempered ductile iron (Etched in picral - 500X)
Table 9, from the original Ductile Iron Society Quality Control Manual, provides a useful summary of heat treatments. Some of the heating times are shorter than generally recommended today for safety against unknown variables.

<table>
<thead>
<tr>
<th>HEAT TREATMENT</th>
<th>Temperature °F and Time</th>
<th>Microstructure</th>
<th>BHN</th>
<th>Grade</th>
</tr>
</thead>
<tbody>
<tr>
<td>1. Hyper-critical Anneal</td>
<td></td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>a. First stage -</td>
<td>1650° - 1750° 1 Hr./In</td>
<td></td>
<td></td>
<td></td>
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<tr>
<td>Carbide breakdown &amp; grain refinement</td>
<td></td>
<td></td>
<td></td>
<td></td>
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<tr>
<td>b. Second stage -</td>
<td>1200°</td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>Furnace cool</td>
<td></td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>c. Third stage -</td>
<td>1300° - 1400° 1 Hr./In</td>
<td></td>
<td>Ferritic</td>
<td>140-180</td>
</tr>
<tr>
<td>Pearlite breakdown</td>
<td></td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>or 35°F/Fr/hr from</td>
<td>1450° - 1200°</td>
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<tr>
<td>2. Sub-critical Anneal</td>
<td></td>
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<td></td>
<td></td>
</tr>
<tr>
<td>Pearlite breakdown</td>
<td>1300° - 1350° 1 Hr./In</td>
<td>Ferritic</td>
<td>140-200</td>
<td>65-45-12</td>
</tr>
<tr>
<td>(Carbide free as-cast)</td>
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<td></td>
<td></td>
<td></td>
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<tr>
<td>3. Stress-Relieving</td>
<td></td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>a. Un-alloyed</td>
<td>950° - 1050° 1 Hr./In</td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>b. Low alloy</td>
<td>1050° - 1100° 1 Hr./In</td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>c. High alloy</td>
<td>1100° - 1250° 1 Hr./In</td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>d. Austenitic</td>
<td>1150° - 1250° 1 Hr./In</td>
<td></td>
<td></td>
<td></td>
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<tr>
<td>Furnace cool a, b, c, d</td>
<td></td>
<td>Very little if any structural change</td>
<td>0-50</td>
<td>60-45-12</td>
</tr>
<tr>
<td>to 550° then air cool</td>
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<td></td>
<td></td>
<td></td>
<td>80-55-06</td>
</tr>
<tr>
<td>4. Normalize &amp; Temper</td>
<td>1650° - 1750° 1 Hr./In</td>
<td>Pearlitic or acicular depending on alloy content.</td>
<td>240-350</td>
<td>100-70-03</td>
</tr>
<tr>
<td></td>
<td>1730° 1 Hr./In then air cool.</td>
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<tr>
<td></td>
<td>800° - 1100° 1 Hr./In</td>
<td></td>
<td></td>
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</tr>
<tr>
<td>5. Quench &amp; Temper</td>
<td>1550° - 1700°F 1 Hr./In</td>
<td>Martensitic</td>
<td>270-350</td>
<td>120-90-02</td>
</tr>
<tr>
<td></td>
<td>Quench in oil or brine. Temper 800° - 1100°F 1 Hr./In</td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>6. Ferritise, Quench &amp;</td>
<td></td>
<td>Ferritic and Martensitic</td>
<td>190-240</td>
<td>80-55-06</td>
</tr>
<tr>
<td>Temper</td>
<td></td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>(1) Reheat to 1400° -</td>
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<tr>
<td>1550°F 4 Hrs.</td>
<td></td>
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<td></td>
</tr>
<tr>
<td>Quench &amp; temper 1200°</td>
<td></td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>1 Hr./In</td>
<td></td>
<td></td>
<td></td>
<td></td>
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<tr>
<td>7. Surface Hardening</td>
<td></td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>a. Induction</td>
<td>Varying (1640°F Min.)</td>
<td>Martensitic</td>
<td>50-62 RC</td>
<td></td>
</tr>
<tr>
<td>b. Flame</td>
<td>Varying</td>
<td>Martensitic</td>
<td>50-62 RC</td>
<td></td>
</tr>
</tbody>
</table>
Compacted graphite or CG iron is related to the ductile iron family. Although it does not have the nodular graphite form characteristic of ductile iron, it is produced by a reduced magnesium treatment to yield controlled vermicular or compacted graphite form with properties between gray iron and ductile iron. Fig. 36 shows typical graphite structure of CG iron.

Compacted graphite iron has strength higher than gray iron, and some ductility, about half that of ductile iron. CG iron with its vermicular graphite form has better thermal conductivity than ductile iron with spherical graphite. Better thermal conductivity makes it more attractive for many heat applications like exhaust manifolds, used widely by European auto makers.

Compacted graphite iron has been chosen for gear pumps and power steering housings because of higher strength than gray iron and better galling resistance than ductile iron.

Other applications traditionally more suitable for gray iron than ductile due to interconnecting flake graphite, are looking at CG iron which offers good thermal conductivity and damping resistance with higher strength than gray iron and some ductility. This combination offers some potential for weight reduction in applications like brake drums and discs, and cylinder blocks and heads, especially for diesel engines. Ingot molds have reportedly experienced longer life in CG iron.

Compacted graphite iron requires the same close controls as ductile iron to maintain closely controlled graphite structure. This requires close control of base composition, sulfur, trace elements, treatment practice, and post inoculation.

If too much elongated flake graphite is obtained, strength and ductility are low. If too much nodular graphite is obtained, then thermal conductivity suffers.

One method of obtaining non spherical compacted graphite form is with closely controlled Mg undertreatment to yield low magnesium residual in the range of .015% .020%, enough magnesium to escape elongated flake graphite but not enough to obtain nodules.
Another treatment method uses a magnesium alloy containing 9% titanium. Titanium is deleterious to spheroidal graphite formation but promotes compacted graphite. Incorporation of titanium residual of .06%-.13% gives more freedom to magnesium control.

Another alloy used in Europe, is a cerium rare earth alloy to yield .02%-.05% cerium which encourages intermediate graphite form.

Another treatment method uses wire feed method of adding magnesium, coordinated with wire feeding of ferrosilicon inoculant, properly balanced to give desirable compacted graphite structure.

In-mold treatment can be used to yield CG iron by adding reduced amounts of regular magnesium alloys in the mold.

The properties of CG iron are intermediate between gray iron and ductile iron as shown in Table 10. CG iron can have a ferritic or pearlitic matrix, providing a range of properties.

**Table 10 - COMPARATIVE PROPERTIES OF IRONS**

<table>
<thead>
<tr>
<th></th>
<th>UTS, ksi</th>
<th>YS, ksi</th>
<th>E%</th>
<th>BHN</th>
</tr>
</thead>
<tbody>
<tr>
<td>Gray Iron</td>
<td>20-50</td>
<td>50</td>
<td>0</td>
<td>140 – 150</td>
</tr>
<tr>
<td>CG Iron</td>
<td>47-75</td>
<td>35-60</td>
<td>1 – 6%</td>
<td>150 - 250</td>
</tr>
<tr>
<td>Ductile Iron</td>
<td>60-120</td>
<td>40-90</td>
<td>2 – 18%</td>
<td>140 - 350</td>
</tr>
</tbody>
</table>

Machinability, damping capacity, and shrinkage are intermediate between gray and ductile iron. Production and control costs are little, if any, lower than ductile iron. But, when lower shrinkage improves yield or property combination permits casting weight reduction, then overall costs may be lower for CG iron parts.

Applications continue to be evaluated where compacted graphite iron might offer a favorable combination of properties for best service.
AUSTEMPERED DUCTILE IRON (A.D. I.)

Austempered ductile iron is a super ductile iron obtained by a specialized heat treatment, quenching in a salt bath or hot oil at a controlled temperature. Strength is doubled while ductility is reduced only slightly. Wear resistance, fracture toughness, and fatigue strength are excellent.

Like ductile iron, austempered ductile iron is a family of high strength irons with varying combinations of properties controlled by the heat treatment. ADI castings have replaced many steel castings and forgings at lower cost in many applications. A popular application is gears. Other applications are crankshafts and connecting rods for high performance engines, spring adapters for rail cars, spring supports in trucks, rolls, track links and guides, etc. Applications are continuing to grow.

Table 11 lists 6 ASTM grades, ranging from 110,000 psi tensile strength with 11% elongation to 200,000 psi strength with 1% elongation and 230,000 psi strength with no elongation specified.

Fig. 37 shows graphically the superior strength-ductility combination of A. D. irons compared to quenched and tempered ductile, ASTM grades of D.I. and gray iron. (15)

Fig. 38 shows how quenching in molten salt cools fast enough to miss the pearlite “hump” and avoid formation of pearlite and ferrite. Then, in the controlled temperature salt bath, isothermal transformation takes place developing a unique mixture of austenite and ferrite called ausferrite.

For the outstanding properties of austempered ductile iron, no special process is necessary in the treatment and casting production. Any well controlled, high quality regular ductile iron can be converted to a chosen grade of austempered ductile iron by a qualified heat treater with the necessary equipment.

Clean, sound castings with good nodularity and high nodule count will yield the desirable properties when heat treated.

The full benefits of the heat treatment process cannot be realized if castings have marginal nodularity, borderline inoculation, microshrinkage, or dross inclusions.

Some thicker castings may be alloyed with nickel and molybdenum to enhance heat treatment response.
Table 11. The Six ASTM Standard ADI Grades (ASTM 897-06)

<table>
<thead>
<tr>
<th>Inch-pound units</th>
<th>Grade 110/70/11</th>
<th>Grade 130/90/09</th>
<th>Grade 150/110/07</th>
<th>Grade 175/125/04</th>
<th>Grade 200/155/02</th>
<th>Grade 230/185/01</th>
</tr>
</thead>
<tbody>
<tr>
<td>Tensile strength, min, ksi</td>
<td>110</td>
<td>130</td>
<td>150</td>
<td>175</td>
<td>200</td>
<td>230</td>
</tr>
<tr>
<td>Yield strength, min, ksi</td>
<td>70</td>
<td>90</td>
<td>110</td>
<td>125</td>
<td>155</td>
<td>185</td>
</tr>
<tr>
<td>Elongation in 2 in., min, %</td>
<td>11</td>
<td>9</td>
<td>7</td>
<td>4</td>
<td>2</td>
<td>1</td>
</tr>
<tr>
<td>Impact energy, ft-lb\textsuperscript{a}</td>
<td>80</td>
<td>75</td>
<td>60</td>
<td>45</td>
<td>25</td>
<td>15</td>
</tr>
<tr>
<td>Typical hardness, HBW, kg/mm\textsuperscript{2}\textsuperscript{b}</td>
<td>241–302</td>
<td>269–341</td>
<td>302–375</td>
<td>341–444</td>
<td>388–477</td>
<td>402–512</td>
</tr>
</tbody>
</table>

\textsuperscript{a}Unnotched charpy bars tested at 72 ± 7°F. The values in the table are a minimum for the average of the highest three test values of the four tested samples.  
\textsuperscript{b}Hardness is not mandatory and is shown for information only.

Fig 37. ADI Properties compared to ductile and gray irons
Obtaining strength-ductility combinations of the selected grade of A.D.I., is accomplished by the heat treater by controlling first the austenitizing temperature and time, then the temperature of the salt bath and the holding time. Lower austempering temperature produces higher strength, hardness and wear with lower ductility. Higher austempering temperature results in higher ductility, fatigue, and impact with relatively lower strength and yield.

Fig 39, graphs the austempering cycles of two grades of ADI. The darker line represents the cycle to produce grade 5, with 200,000 psi tensile strength, 1% elongation, and Brinell hardness 400-500. Castings are austenitized at 1700F for 1 ½ hours, then quenched into a molten salt bath at 450F and held 2 ½ hours maximum.

The lighter line represents a cycle for grade 1, with strength of 125,000 psi and the highest elongation of 10%, with a hardness range of 260-320 BHN. Castings are austenitized at 1500F for 1 hour maximum, then quenched into the salt bath at 750F and held for 2 ½ hours maximum.

Fig 40, shows a typical microstructure of an austempered ductile iron at 500 magnification and 5000 magnification.

Some advantages reported for A.D.I. castings are: (16)
- Lighter weight possible, up to 10%
- Damping 5 times steel
- Thermal conductivity higher than steel
- Good machinability for high strength levels
- Manufacturing flexibility, nearer net shape
- Lower machining cost

The most successful applications of ADI have been replacements for steel forgings providing high performance at a cost 20% to 40% less than forgings. (16)

Applications continue to grow for austempered ductile iron. The best potential markets can be found in the 1 ½ million tons of forgings imported from off shore.
Some fields of applications have been:

- Gears for many applications
- Crankshafts - for high performance autos, diesel engines, compressors
- Engine parts - connecting rods, camshafts
- Vehicles - suspension spring seats and brackets, yokes, selector forks, steering components
- Off highway vehicles - track links and guides, suspension arms and sprockets
- Agricultural equipment - planting tool, plow shares and tips
- Railway components - brake blocks and shoes, suspension links and cradles
- Cutting tools – shredders, milling cutters
- Rolls - for wire mill, conveyor systems
- Miscellaneous - slide bearings, wear guides, hydraulic motor components
APPENDIX VI

AUSTENITIC (NI RESIST) AND ALLOY DUCTILE IRONS

AUSTENITIC DUCTILE IRONS

High nickel gray irons known as Ni Resists* have been used for many applications that require high resistance to erosion, corrosion, heat and oxidation. The 18% to 36% nickel contents retain a more stable austenitic matrix that does not transform to pearlite or ferrite.

With the discovery of magnesium treatment to produce nodular graphite form, this treatment was applied to the austenitic irons to obtain better properties from nodular graphite form.

As a result, a family of austenitic ductile irons was developed with nickel contents ranging from 18% to 36%. Some grades include chromium contents ranging from 2% to 5%, others with high manganese 1.80% to 4.5% and two grades with silicon in the 5.5% range.

Molybdenum of 1.00X concentration may be added to two grades for higher hot strength. Table 12 lists 11 grades of austenitic ductile irons. Table 13 lists compositions and basic properties. (3)

Tensile strengths range 58,000 to 85,000 psi, yield strengths 28,000 to 45,000 psi, and elongations of the chromium containing grades range 6% to 20%, and the grades without chromium range 20% to 40%. Fig. 41 shows microstructure of D-2 ductile Ni Resist.

To obtain the strength and toughness advantages of a nodular graphite form requires the same controlled magnesium addition. With the high nickel content, nickel magnesium treatment alloys can be used and magnesium control is easier. However, base composition control is more complex, charging various alloy returns and alloys to obtain the complex alloy compositions for the specified grades.

Some special foundry techniques are used on the Ni Resist grades. And special melting precautions are needed to reduce the increased tendency for absorption of hydrogen and nitrogen gasses. The metal should not be overheated or held unnecessarily long. Chromium and manganese alloys should not be melted down with the charge but added late in the heat.

*International Nickel Co. trade name for austenitic iron.
Table 12. Chemical Compositions of Austenitic Ductile Irons***

<table>
<thead>
<tr>
<th>Type Designation</th>
<th>C%</th>
<th>Si%</th>
<th>Mn%</th>
<th>Ni%</th>
<th>Cr%</th>
<th>P%</th>
</tr>
</thead>
<tbody>
<tr>
<td>D-2**</td>
<td>3.0 max.</td>
<td>1.75 — 3.00</td>
<td>0.70 — 1.00</td>
<td>18.0 — 22.0</td>
<td>1.75 — 2.50</td>
<td>0.08 max.</td>
</tr>
<tr>
<td>D-2B</td>
<td>3.0 max.</td>
<td>1.75 — 3.00</td>
<td>0.70 — 1.00</td>
<td>18.0 — 22.0</td>
<td>2.75 — 4.00</td>
<td>0.08 max.</td>
</tr>
<tr>
<td>D-2C</td>
<td>2.9 max.</td>
<td>2.00 — 3.00</td>
<td>1.80 — 2.40</td>
<td>21.0 — 24.0</td>
<td>0.50 max.</td>
<td>0.08 max.</td>
</tr>
<tr>
<td>D-2M</td>
<td>2.7 max.</td>
<td>1.50 — 2.60</td>
<td>3.75 — 4.50</td>
<td>21.0 — 24.0</td>
<td>0.50 max.</td>
<td>0.08 max.</td>
</tr>
<tr>
<td>D-3***</td>
<td>2.6 max.</td>
<td>1.50 — 2.80</td>
<td>0.50 max.</td>
<td>28.0 — 32.0</td>
<td>2.50 — 3.50</td>
<td>0.08 max.</td>
</tr>
<tr>
<td>D-3A</td>
<td>2.6 max.</td>
<td>1.50 — 2.80</td>
<td>0.50 max.</td>
<td>28.0 — 32.00</td>
<td>1.00 — 1.50</td>
<td>0.08 max.</td>
</tr>
<tr>
<td>IN 854</td>
<td>2.0</td>
<td>5.5</td>
<td>0.60</td>
<td>36.0</td>
<td>2.0</td>
<td>0.08 max.</td>
</tr>
<tr>
<td>D-4</td>
<td>2.6 max.</td>
<td>5.0 — 6.0</td>
<td>0.50 max.</td>
<td>28.0 — 32.0</td>
<td>4.50 — 5.50</td>
<td>0.08 max.</td>
</tr>
<tr>
<td>D-5</td>
<td>2.4 max.</td>
<td>1.50 — 2.75</td>
<td>0.50 max.</td>
<td>34.0 — 36.0</td>
<td>0.10 max.</td>
<td>0.08 max.</td>
</tr>
<tr>
<td>D-5B</td>
<td>2.4 max.</td>
<td>1.50 — 2.75</td>
<td>0.50 max.</td>
<td>34.0 — 36.0</td>
<td>2.0 — 3.0</td>
<td>0.08 max.</td>
</tr>
<tr>
<td>Nodumag</td>
<td>3.0 max.</td>
<td>2.00 — 3.00</td>
<td>6.0 — 7.0</td>
<td>12.0 — 14.0</td>
<td>N.S.</td>
<td>N.S.</td>
</tr>
</tbody>
</table>

Symbols:
* Including incidental cobalt.
** Addition of 0.7 — 1.0% molybdenum increases strength above 800°F (425°C).
*** Magnesium content necessary is the same as in any other ductile iron.
N.S. Not Specified.

Fig 41. Ni Resist D-2, 250X Etched
## Composition and mechanical properties of NI resist Austenitic Ductile Irons

<table>
<thead>
<tr>
<th></th>
<th>D2</th>
<th>D2-B</th>
<th>D-2C</th>
<th>D-2M</th>
<th>D-3</th>
<th>D-3A</th>
<th>D-4</th>
<th>D-5</th>
<th>D-5B</th>
</tr>
</thead>
<tbody>
<tr>
<td>C, max</td>
<td>3.00</td>
<td>3.00</td>
<td>2.90</td>
<td>2.70</td>
<td>2.60</td>
<td>2.60</td>
<td>2.60</td>
<td>2.40</td>
<td>2.40</td>
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<tr>
<td>Si</td>
<td>1.75-3.00</td>
<td>1.75-3.00</td>
<td>2.0-3.00</td>
<td>1.50-2.50</td>
<td>1.50-2.80</td>
<td>1.50-2.80</td>
<td>5.0-6.0</td>
<td>1.50-2.75</td>
<td>1.50-2.75</td>
</tr>
<tr>
<td>Mn</td>
<td>0.70-1.0</td>
<td>0.70-1.0</td>
<td>1.80-2.40</td>
<td>3.75-4.50</td>
<td>0.50 max</td>
<td>0.50 max</td>
<td>0.50 max</td>
<td>0.50 max</td>
<td>0.50 max</td>
</tr>
<tr>
<td>P</td>
<td>0.08 max</td>
<td>0.08 max</td>
<td>0.08 max</td>
<td>0.08 max</td>
<td>0.08 max</td>
<td>0.08 max</td>
<td>0.08 max</td>
<td>0.08 max</td>
<td>0.08 max</td>
</tr>
<tr>
<td>Ni</td>
<td>18.0-22.0</td>
<td>18.0-22.0</td>
<td>21.0-24.0</td>
<td>21.0-24.0</td>
<td>28.0-32.0</td>
<td>28.0-32.0</td>
<td>29.0-32.0</td>
<td>34.0-36.0</td>
<td>34.0-36.0</td>
</tr>
<tr>
<td>Cr</td>
<td>1.75-2.50</td>
<td>2.75-4.0</td>
<td>0.50 max</td>
<td>0.20 max</td>
<td>2.50-3.00</td>
<td>1.00-1.50</td>
<td>4.50-5.50</td>
<td>0.10 max</td>
<td>2.00-3.00</td>
</tr>
<tr>
<td>UTS, ki</td>
<td>58-60</td>
<td>58-70</td>
<td>58-65</td>
<td>70-85</td>
<td>55-65</td>
<td>55-65</td>
<td>60-70</td>
<td>55-60</td>
<td>55-65</td>
</tr>
<tr>
<td>E%</td>
<td>8-20</td>
<td>7-15</td>
<td>20-40</td>
<td>40-50</td>
<td>6-15</td>
<td>10-20</td>
<td>-</td>
<td>20-40</td>
<td>6-12</td>
</tr>
<tr>
<td>BHN</td>
<td>139-202</td>
<td>148-211</td>
<td>121-171</td>
<td>139-202</td>
<td>139-202</td>
<td>121-193</td>
<td>202-273</td>
<td>131-185</td>
<td>139-193</td>
</tr>
</tbody>
</table>

### Charpy V-notch Impact Ft Lbs

<table>
<thead>
<tr>
<th></th>
<th>Room T</th>
<th>0 F</th>
<th>-100 F</th>
<th>-320 F</th>
<th>Proportional limit, ksi</th>
<th>Modulus of elasticity psi X 10e6</th>
</tr>
</thead>
<tbody>
<tr>
<td>Room T</td>
<td>12.5</td>
<td>-</td>
<td>-</td>
<td>-</td>
<td>16.5-18.5</td>
<td>16.5-18.5</td>
</tr>
<tr>
<td>0 F</td>
<td>11.5</td>
<td>-</td>
<td>-</td>
<td>-</td>
<td>16-19</td>
<td>16.5-18.5</td>
</tr>
<tr>
<td>-100 F</td>
<td>10.0</td>
<td>-</td>
<td>-</td>
<td>-</td>
<td>12-16</td>
<td>16-19</td>
</tr>
<tr>
<td>-320 F</td>
<td>4.5</td>
<td>-</td>
<td>-</td>
<td>-</td>
<td>12-16</td>
<td>12-16</td>
</tr>
<tr>
<td>Proportional limit, ksi</td>
<td>16.5-18.5</td>
<td>16-19</td>
<td>16.5-18.5</td>
<td>16.0-18.5</td>
<td>16.0-20.0</td>
<td>16.0-17.5</td>
</tr>
</tbody>
</table>
Table 14  SOME PROPERTIES AND APPLICATIONS OF AUSTENITIC D.I. GRADES

<table>
<thead>
<tr>
<th>Grade</th>
<th>Attractive Properties</th>
<th>Applications</th>
</tr>
</thead>
<tbody>
<tr>
<td>D-2</td>
<td>Good resistance to corrosion.</td>
<td>Pump and impellers, valve, parts.</td>
</tr>
<tr>
<td>D-2B</td>
<td>Erosion and frictional wear, Heat resistance up to 1400F</td>
<td>Pulp grinding</td>
</tr>
<tr>
<td>D-2C</td>
<td>Higher ductility, can be welded, lower heat and corrosion resistance</td>
<td></td>
</tr>
<tr>
<td>D-2M</td>
<td>Good low temperature properties down, to -320F for cryogenic applications</td>
<td>Pumps, valves and compressors handling liquified gases</td>
</tr>
<tr>
<td>D-3</td>
<td>Good high temperature properties, resistance to erosion or steam and wet slurries</td>
<td>Pump impellers for steam applications, corrosive slurries</td>
</tr>
<tr>
<td>D-3A</td>
<td>Resistant to wear and galling</td>
<td>Gas turbine components</td>
</tr>
<tr>
<td>D-4</td>
<td>Higher resistance to oxidation and corrosion, Poorer machinability</td>
<td>Turbo charger and gas turbine applications up to 1500F</td>
</tr>
<tr>
<td>D-5</td>
<td>Minimum thermal expansion. Excellent ductility</td>
<td>Glass molds, machine tool parts</td>
</tr>
<tr>
<td>D-5B</td>
<td>Low thermal expansion</td>
<td>Low expansion applications requiring more resistance to oxidizing and high temperature</td>
</tr>
</tbody>
</table>

The International Nickel Co. publishes bulletins with details of compositions, properties and service applications of all grades of Ni Resist. Their guidance is recommended for any foundry considering production or these grades.

ALLOYED ACICULAR DUCTILE IRONS
Another member of the ductile iron family is an alloy of approximately 3% nickel and .50%-1.0% molybdenum. Nickel is increased for heavier sections. These alloys increase hardenability so that an acicular bainitic structure is obtained as-cast, without heat treatment. Fig. 42 shows the acicular bainitic structure. Similar to properties of a quenched and tempered structure, tensile strengths, as-cast, range up to 200,000 psi with yield strengths up to 170,000 psi and elongations 1%-3%.
HIGH SILICON DUCTILE IRON FOR HEAT RESISTANCE
An inexpensive ductile iron with high silicon contents in the 4% to 6% range and molybdenum of .50-2.0% has provided good high temperature service in temperatures up to 1500°F in many applications.

High silicon contents cause low impact at low temperatures, but in high temperature applications the high silicon level raises the transformation temperature to 1500 and 1600°F as silicon is increased.

In alternating heat cycles up to 1200-1500°F regular ductile iron goes through repeated growth transformations giving poor thermal fatigue, resulting in cracking and oxidation.

With high silicon contents of 4%-6% and transformation temperature raised, cyclic transformations do not occur and better thermal shock resistance results. Molybdenum increases hot strength and further improves the performance of these alloys.
APPENDIX VI I

EFFECTS OF COMMON ELEMENTS

CARBON is the most important and most abundant element in ductile iron.

In ductile irons, carbon contents may range from 3.30% to 3.90%. In molten iron, this carbon is in solution. As the iron solidifies, most of this carbon must come out of solution and is precipitated as graphite particles. When properly treated with magnesium, these graphite particles precipitate as spheroids or nodules which provide ductility and higher strengths than the flake form of graphite in gray iron.

Some carbon may remain in a combined form as pearlite, depending on the balance of graphitizing elements against stabilizing elements, and the cooling rate.

High carbons increase the urge to precipitate graphite and reduce the tendency to form brittle carbides. High carbons of 3.60-3.80% are desirable in thin sections.

High carbon contents are more readily treated to nodular graphite under marginal condition.

High C contents increase the momentum of graphitization and encourage a more ferritic matrix. High carbons increase fluidity, castability, and machinability, and reduce shrinkage.

In pipe centrifugally cast on metal molds, carbons around 3.50% are preferred for better surface.

In heavy sections, and pearlitic grades, lower carbons in the 3.30-3.60% range may be preferred.

The best carbon level should be chosen for the castings being produced. Then, for best control and minimum variability, carbon should be controlled within a 0.20% range and preferably within a 0.15% spread.

It must be remembered that all reference to carbon content means final carbons after treatment. Because a carbon drop of 0.10% to 0.15% occurs in most treatment processes, the furnace carbon before treatment must be higher. For ferritic thin castings, furnace preliminary carbons are generally targeted in the 3.75%-3.90% carbon range.

High carbon content is generally beneficial to most desires, but with excessively high final carbons, a graphite flotation defect can be encountered.

The excess carbon as graphite nodules tend to float out and concentrate in the top of heavy sections giving poor properties and surface.

Thin sections solidify fast enough to avoid any flotation opportunity.

The risk of flotation defect is greatest in heavy sections when carbon exceeds 3.75% or carbon equivalent of C+1/3 Si exceeds 4.55%.
SILICON
Next to carbon, silicon is a most important element. Silicon is a graphitizer adding to the effect of carbon in encouraging graphitization, preventing carbides, and encouraging a ferritic matrix. Silicon also increases the strength of ferrite, contributing to a high combination of strength and ductility.

Nucleation for good graphite form and size is accomplished by late additions of ferrosilicon, containing calcium and aluminum residuals.

For ferritic small castings, final silicon contents usually are targeted in the range of 2.50% to 2.80%.

However, high silicon is unfavorable to impact properties and ductile-to-brittle transition temperature.

For applications where service may be in cold climates and impact strength is important, silicon maximum of 2.50% is specified as necessary to meet impact test requirements. In these applications, final silicon is usually targeted in the 2.20%-2.50% range and carbon may be raised about .10%. Very low phosphorus content below .02% improves impact and can permit higher silicon maximum.

Silicon range should be selected for the application and specification involved. For best control and minimum variability, final silicon should be targeted within 0.30% range and preferably with a 0.20% spread.

To allow for silicon from the treatment alloy and post inoculation, furnace silicon is usually targeted 1.20%-1.50% lower than final silicon target.

CARBON EQUIVALENT
Carbon equivalent is a most significant single factor combining the total graphitizing effects of carbon and silicon.

The simplest formula that best reflects solidification arrest and graphitizing influence is C\text{Eq} = C + \frac{1}{3} Si.

Other elements have influence but since they vary little this simple formula provides the most useful guidance.

Chemical composition should be targeted toward a consistent carbon equivalent range. If carbon is on the high side of desirable range, then silicon should be targeted toward the low side in order to maintain a consistent carbon equivalent.

High side silicon along with high side carbon would yield too high a carbon equivalent, out of good control range.

For shrinkage consideration silicon was found less influential, and the best shrinkage formula is C\text{Eq} = C + \frac{1}{7} Si, with above 3.9% desirable. Shrinkage decreases as carbon equivalent increases.

For ferritic small castings, carbon equivalent range of 4.45%-4.55% is popular.
For pearlitic grades and possibly for heavy sections, there may be reason for lower carbon equivalent.

Carbon equivalent range should be selected for the castings produced and for best uniformity should be controlled within a 0.15% spread, and preferably within a 0.10% range.

MANGANESE
In gray iron, manganese is desirable and is added to keep manganese in a favorable ratio to sulfur. In steels, manganese serves a useful purpose and is usually present in ranges of .30 to .70%.

But, in ductile iron, manganese is not needed, is not added, and lowest possible concentration of .30% or lower is desirable in ferritic grades.

Since manganese stabilizes pearlite, higher concentration of .40 to .70% can be tolerated in pearlitic grades as part of the total alloy combination needed.

Manganese in higher concentrations can increase carbide tendency.

Manganese is one of the elements that have an unfavorable tendency to segregate toward grain boundaries. In the slow solidification of heavy sections these intercellular segregates can become detrimental to overall properties.

The manganese level chosen should be controlled within a 0.10% range.

SULFUR
Sulfur, an element present in gray iron and steels, is an element to be eliminated in producing ductile iron. Removing sulfur to negligible levels seems necessary to the nodularizing process. Nodulizing elements like magnesium, first react with and remove sulfur. Roughly, one pound of magnesium is required to remove one pound of sulfur. Varying sulfur content complicates the control of magnesium residual needed for consistent nodularity.

For that reason, low sulfur base iron below .02% is desirable for most treatment processes and below .01% for in-mold treatment.

Base sulfur should be controlled

- within a 0.010% range for ladle treatment
- within a 0.005% range for in-mold treatment.

After treatment with magnesium, spectrometer analysis usually reports sulfurs of .005 to .015%, which content is mostly very fine magnesium sulfide inclusions.

If high sulfur base iron is treated, spectrometer sulfur content is likely to run higher and magnesium analysis is likely to contain more magnesium sulfide inclusions and less active magnesium. Slightly higher magnesium contents are usually necessary to insure good nodularity.

After good quality nodular iron has been produced and cast, it can be contaminated in the mold by high sulfur sands. Absorption of sulfur from the sand or facing can cause
reversion to flake graphite on the surface. Sulfur in the sand should be controlled below 0.015%.

PHOSPHORUS
In gray iron, phosphorus causes complications above 0.15% P. Phosphorus first forms brittle phosphides detrimental to ductility and machinability. In higher concentrations, a low melting eutectic causes porosity and leakage at section changes and hot spots.

In ductile iron, phosphorus is detrimental to ductility and lower phosphorus contents below 0.05% are necessary to obtain the potential ductility of ductile iron.

For best impact properties, the lowest possible phosphorus content is desired, below 0.03%. Each 0.01% reduction in phosphorus, favorably lowers impact transition temperature by 10 degrees F.

MAGNESIUM
Magnesium has proven to be the most practical nodulizing element. After loss to vaporization, the first magnesium desulfurizes and deoxidizes before any residual is available for nodulizing.

A net retained residual of 0.03 to 0.05% magnesium usually produces a fully nodular graphite structure.

With high sulfur base iron, slightly higher magnesium residuals may be required because more of the magnesium seen by the Spectrometer is magnesium sulfide rather than active magnesium. Also, heavy sections may require slightly higher magnesium residuals.

When magnesium is below the necessary minimum, some vermicular graphite results and properties are lower.

With low magnesium levels of 0.015 to 0.020%, compacted graphite iron is intentionally produced.

High magnesium contents above 0.06% are undesirable because of increased risk of carbides and increased dross tendency and shrinkage.

Magnesium content chosen for the castings produced should be controlled within a 0.015% range.

CERIUM AND RARE EARTHS
Cerium has become a regular supplementary element in ductile iron production because of its protective value against detrimental trace elements.

The first ductile iron was made with cerium as nodulizer at a residual level of 0.035% in a very hyper eutectic iron. But, as a nodulizer, cerium is not as flexible and practical as magnesium.

In addition to protecting against and raising the safe limits of several detrimental tramp elements, cerium assists the magnesium in desulfurizing, deoxidizing, and nodulizing.

Excessive cerium with adequate magnesium increases carbide tendency.
Magnesium treatment alloys are available with several levels of cerium (or rare earths) and consistent cerium control is important along with magnesium. It is desirable that cerium be included in Spectrometer analysis. Most foundries like a final cerium content of .005% to .01%.

Cerium is the principal element (usually about 45%) in mischmetal which contains other members of the rare earth family like lanthanum, neodymium, praseodymium, samarium and yttrium.

These other members of the rare earth family, though not as effective, assist cerium in neutralizing tramp elements, desulfurizing, and assisting nodularity.

Treatment alloy should be purchased and used with full knowledge of cerium and rare earth content.
APPENDIX VIII

TRACE ELEMENTS AND ALLOYS

TRACE ELEMENTS SUBVERSIVE TO GRAPHITE NODULARITY

LEAD (Pb)
Lead is one of the most potent subversive elements. Slightest traces of lead at .002% and higher can cause irregular, flake, and lacy graphite. Cerium can increase tolerance some, but lead is a most dangerous contaminant.

Fig. 43 shows vermicular graphite from .003% lead.

Lead may be introduced from paint, bearings, brass, solder, leaded free machining steels, or from some fluorspar sources.

ANTIMONY - (Sb)
Antimony above .002% and especially above .004% is detrimental to graphite nodularity, and also stabilizes pearlite. Antimony also segregates strongly in cell boundaries. Vitreous enamel, paint, bearings, and low melting pot metal can introduce antimony.

BISMUTH (Bi)
Bismuth above .002% also is detrimental to graphite nodularity especially in heavy sections.

Europeans report that traces of bismuth when properly neutralized with cerium, increase nodule count.

Most malleable iron contains a trace addition of bismuth. Type metal is predominantly bismuth.

TITANIUM (Ti)
Titanium improves the flake graphite structure of gray iron, but is detrimental to ductile iron nodularity above .05%, especially in heavy sections. Fig. 44 shows poor graphite from .075% titanium. Titanium can be introduced from some pig irons, or gray irons, and some paint.

TELLURIUM - (Te)
Small additions of tellurium up to .02% have been used in gray iron and some in ductile iron to eliminate pin holes. But in ductile iron, tellurium is detrimental to nodularity as well as casting surface, and should be controlled below .02%. Fig. 45 shows very poor graphite from .05% tellurium.
Deteriorated graphite from excessive trace elements

Fig 43. 0.003% Lead (vermicular & flake)  
Fig 44. 0.075% Titanium (spheroids with attached flakes)

Fig 45. 0.05% Tellurium (vermicular graphite)  
.13% Aluminum (vermicular graphite)

ZINC (Zn)  
Zinc is an increasing threat because increasingly more automotive steel is being galvanized.

In cupola melting, most of the zinc is volatilized, complicating the effluent disposal, but leaving little detriment to the iron.

In induction melting, zinc has penetrated the lining and damaged coils. In Europe, troubles have been experienced from graphite deterioration from excessive zinc in the iron.

Much is yet to be learned as threats of zinc contamination increase.
ALUMINUM - (AI)
Aluminum is present in trace amounts in alloys and is beneficial in inoculants. These do not cause excessive concentration unless supplemented by other inadvertent sources. But, if all cumulative sources of aluminum exceed .04%, graphite nodularity can be damaged.

Fig. 46 shows deteriorated graphite from .13% aluminum.

Aluminum is present in killed steels, and aluminum metal can be attached to steel scrap.

ZIRCONIUM (Zr)
Zirconium above .01% can distort nodular graphite shape.

SELENIUM (Se) similar to sulfur, causes flake graphite and should be below .01%.

Combinations of these trace elements can be additive in effect. However, cerium can precipitate out and counteract marginal amounts of these deleterious elements and extend safe concentration levels. However, it is safer to avoid these contaminating elements than to depend on cerium protection.

There is yet much to be learned about trace element combinations and their neutralization.

UNDESIRABLE CARBIDE FORMERS

BORON - (B)
Boron is a most powerful carbide former at trace levels of .002% and must be avoided.

Boron can be absorbed from new induction furnace linings with borate binder. Other sources can be vitreous enamels, some steels, malleable iron scrap, etc.

CHROMIUM - (Cr)
Chromium is a very potent carbide and pearlite stabilizer.

In ferritic grades, chromium should be kept below .05%.

In pearlitic grades, chromium may be tolerated up to .10%.

Beyond .10%, chromium increases carbide tendency strongly and retards annealing.

For wear and abrasion resistance, chromium is intentionally added to produce some wear resistant carbides in the microstructure.

In some grades of austenitic ductile iron Ni Resist, chromium is specified to obtain heat and wear resistant carbides.

Sources of inadvertent chromium are alloy steels, chrome plated steels, stainless steels, high chromium wear resistant irons, etc.

VANADIUM - (Va)
Vanadium, like chromium, stabilizes carbide and pearlite. Maximum level for ferritic grades should be .02% and for pearlitic grades .05%.
Vanadium may come from inadvertent alloy steels, or tool steels in scrap.

PEARLITE FORMERS
Several alloys which do not stabilize carbide, but do stabilize pearlite are useful in producing pearlitic high strength grades but must be avoided in ferritic high ductility grades.

COPPER (Cu)
Copper is a favorite alloy to encourage pearlite because it is not a carbide former.

High purity electrolytic copper should be used. Secondary copper can be contaminated with lead, arsenic, antimony, and hydrogen.

For 80-55-06 grade, copper additions of .30 to .50% are usually made depending on the concentration of manganese and trace alloys, to obtain a 40% to 60% pearlite matrix.

For 100-70-03 grade, copper additions of .50 to 1.00% are usually made to produce a matrix of 80% to 100% pearlite.

Manganese, as described under regular elements, also stabilizes pearlite. Manganese additions are not recommended, since copper is preferable, but manganese level is a part of the alloy combination factor which determines the amount of copper needed for a certain proportion of pearlite.

TIN - (Sn)
Tin, like copper, stabilizes pearlite but does not promote carbides. Tin is very powerful, ten times as potent as copper. As little as .05% to .10% tin can yield a fully pearlitic matrix, and as little as .03% to .05% tin can yield enough pearlite for 80-55-06 grade.

In ferritic grades, tin should be held below .01%.

Intentional use of tin requires good spectrometer calibration and attention.

ARSENIC - (As)
Arsenic, like tin, is a powerful pearlite stabilizer. As little as .05% will yield a fully pearlitic matrix.

In ferritic grades, arsenic should be less than .01%.

ALLOYS FOR STRENGTH - HARDENABILITY
NICKEL (Ni)
Nickel is a useful alloy in various ductile iron families. Below 1%, nickel is a graphitizer and strengthens ferrite, consequently increasing tensile and yield strength with little reduction in elongation.

Above 1%, nickel begins to stabilize pearlite.

In high strength acicular grades, nickel additions of 3% to 8%, with molybdenum, increase hardenability, making possible the acicular bainitic structure and its outstanding properties.
In the austenitic Ni Resist grades, nickel concentrations of 18% to 36% provide the austenitic structure with exceptional properties along with erosion, corrosion, and heat resistant properties.

**MOLYBDENUM - (Mo)**
Molybdenum in concentrations of 0.50% to 2.00% assist nickel to obtain the properties in acicular ductile irons.

In lower concentrations, molybdenum promotes pearlite.

In ferritic grades, molybdenum should be held below .03%.

**GASEOUS CONTAMINATING ELEMENTS**
As described in Chapter 3, excessive absorption of nitrogen and hydrogen can cause mysterious troubles.

**Nitrogen** at low concentration contributes some strength and pearlite stabilization. At higher concentrations, carbide tendencies increase. Very high concentrations are expelled upon solidification and can form gas holes.

Nitrogen usually causes no problem at concentrations below .0040% or 40 parts per million but can cause problems above .010" or 100 parts per million.

Nitrogen is absorbed from some alloys, some chemical no-bake binders, and from electric arc furnaces.

**Hydrogen** at very low concentrations increases carbide tendency. When hydrogen segregates and concentrates in the center of a section, "center line carbides" or "inverse chill" can result. Very high hydrogen concentrations are also expelled upon solidification causing subsurface round gas holes.

Hydrogen usually causes little problem at concentrations of .0002% or 2 parts per million but can cause problems around .0004% or 4 parts per million.

After magnesium treatment, hydrogen is readily absorbed from damp alloys, newly lined ladles or wet patches insufficiently heated or from high moisture green sand.

* * * *

Much is yet to be learned about contaminants and trace elements. But, enough is known to emphasize that highest quality ductile iron is a technical challenge requiring the knowledge and closest control of many variables and contaminants.
APPENDIX IX

IMPACT STRENGTH - FRACTURE TOUGHNESS

One attractive property of ductile iron is ductility, the ability to bend and stretch as measured by percent elongation in the slow loaded tensile test.

An equally attractive property of ductile iron is impact strength, the ability to resist the shock of high velocity stresses. This impact property is more difficult to test and measure because it is influenced by temperature, velocity of impact, size of specimen, and the effect of design angles, casting flaws, etc.

Good service performance of many ductile iron parts verifies its serviceable impact resistance. Many ductile iron castings in service have withstood shock loads that caused failure of gray iron parts.

Ductile iron pipe have admirably withstood earthquakes and the "water hammer" impact of valve closings.

But, laboratory testing of impact qualities has been more difficult.

CHARPY IMPACT TEST
An early and popular impact test is the Charpy test in which a specimen is broken by a swinging pendulum and energy absorbed in breakage calculated. Small machined specimens are tested either unnotched or with a special V notch. Impact strength is calculated in foot pounds or in Joules and various metric designations in different countries.

The Charpy test is easiest to perform, but a disadvantage is the specimen too small to represent service performance and the full section.

TRANSITION TEMPERATURE
Above a certain temperature, irons and steels break with a ductile fracture. Upper shelf energy is higher and increases little with further increases in temperature.

Below a certain low temperature, the fracture is a brittle cleavage fracture and the energy level is much lower. The midpoint temperature where half the fracture is ductile (gray) and half brittle cleavage (white crystalline) is considered the transition temperature.

In cold climates, a low transition temperature is desirable where impact is anticipated.

CHARPY V NOTCH IMPACT STRENGTHS
Fig. 47, from Research Report #15, shows a Charpy V-notch curve for ferritic ductile iron compared to cast steel.

By this test, the upper shelf impact energy of ductile iron is about 10-12 ft. lbs. The transition temperature is approximately - 40F and the lower shelf impact energy is about 4 ft. lbs.

The cast steel has a much higher upper shelf impact energy, but a much higher and more gradual transition temperature.
Actually between -30F and +20F ductile iron shows slightly higher impact strength.

In ductile iron the highest impact energy values of 10-15 ft. lbs. are obtained with a fully ferritic matrix.

A subcritical anneal provides the best impact properties with the highest upper shelf energy and the lowest transition temperature.

Military specification Mil-1-2243 requires a Charpy V-notch impact of 10 ft. lbs at 20F. And International I.S.O standard 370-17 requires 9.5 ft. lbs.

Decreasing silicon content improves impact energy and transition temperature. Several specifications limit silicon to 2.50% maximum on high ductility grades where impact loading may be encountered.

Decreasing phosphorus content below .03% improves impact energy and transition temperature. For each phosphorus decrease of .01%, impact transition temperature is favorably lowered 10 degrees F.

Pearlite increases progressively decrease impact energy and increase transition temperature.

High strength, quenched and tempered ductile irons have Charpy V impact values of approximately 5 ft. lbs and unnotched values of 25-40 ft. lbs. with lower transition temperatures than pearlitic irons of similar strength.

High strength alloyed acicular ductile irons have Charpy V values of 4 to 6 ft. lbs at very high strength levels.

Austenitic ductile iron Ni-resist grades have very good impact strengths at low temperatures. The low chromium grades range from 10 to 25 ft. lbs Charpy V at -100 F.
Experts in fracture mechanics are generally preferring the drop weight dynamic tear test for more useful indication of impact resistance and flaw tolerance.

Larger equipment is required to test larger specimens 1 5/8 in. X 7 1/8 in. X 5/8" thick. The sawed notch is much sharper.

Fig. 48, from Research Project #15, shows fracture toughness $K_{1c}$ values from this test, compared with cast steel.

Cast steel again showed higher upper shelf values but higher transition temperature than ductile iron. In this test, ductile iron with 2.20% silicon showed greater superiority over a wider and more serviceable range of temperature from -30F to +70F.
Dynamic tear tests on ferritic 60-40-18 grade gave $K_{1c}$ values around 80 ksi which indicates very good flaw tolerance.

In pearlitic grades $K_{1c}$ values were lower around 30 ksi, which is not as flaw tolerant.

Research is continuing toward a better understanding of fracture mechanics and more useful impact tests.
Cyclic loading is a common cause of structural failure.

Fatigue strength or limit is the level of strength that a specimen will resist failure through an unlimited number of cyclic stresses, usually 1 million to 10 million cycles. Cycles under 500,000 are considered short cycle fatigue tests.

Janowak, Alagarsamy, and Venugopalan reported useful fatigue data on commercial ductile irons from three foundries using a Moore type rotating bending machine. (19)

Fig. 49 is one of their graphs representing iron 5 from foundry B, which is an as-cast pearlitic grade, ranging in tensile strengths from 96,000 to 110,000 psi, yield strengths from 58,000 to 67,000 psi, and elongations from 5% to 12%.

This graph portrays the scatter obtained within the grade and the trends of declining fatigue stress as test cycles were increased from 100,000 cycles to 1 million, and on to 10 million cycles.

Another popular fatigue test is the Wohler rotating bending test using a .417 inch diameter machined specimen, tested either unnotched or with a circumferential 45 degree V-notch.

Endurance or fatigue ratio is the ratio of fatigue strength to tensile strength. In carbon and low alloy steel castings, the fatigue ratio usually ranges 43 to 48% of the tensile strength.

Unnotched fatigue tests on ferritic ductile iron usually average about 35,000 psi or 50% of the tensile strength.

Unnotched pearlitic ductile irons average about 40,000 psi or 45% of the tensile strength.

Fatigue ratio of notched ferritic ductile specimens is about 63% of the unnotched specimens, or 31% of the tensile strength.

The fatigue ratio of notched pearlitic specimen is about 60% of the unnotched specimen or 24% of the tensile strength.

Ferritic grades have a higher fatigue ratio, but higher strength pearlitic grades have overall higher fatigue strength and are usually preferred where high fatigue strength is desired.

Rough surfaces decrease fatigue strength and shot peening improves fatigue strength.
It can be expected that in future years experience and research will continue to add to our knowledge of the properties of the many ductile irons, and their applications will be extended.

Continued improvement in quality controls and quality consistency will increase confidence in further uses of ductile irons.
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