MANUFACTURING OF NODULIZING ALLOYS AND THEIR APPLICATION

Michele Sarlo - Italmagnésio S/A Ind. e Com. São Paulo - Brazil

Helmut Richter - Italmagnésio S/A Ind. e Com. Minas Gerais - Brazil

Italmagnésio S/A
Rua Francisco Tramontano, 100
05686 - São Paulo - SP - Brasil

ABSTRACT

A great technological development has provided substantial improvements in the quality of nodulizing alloys, resulting in a smaller percent of addition of this material in the base iron, with the following advantages:

- decrease in slag formation
- smaller amount of silicon introduced, making possible the addition of larger quantities of post-inoculants and/or the use of a larger quantity of returns in the base iron.

The main objective of this work is to show the manufacture and use of FeSiMg alloy of high quality, enabling the user to obtain the stated advantages.

The manufacturing process of FeSiMg alloys is discussed, covering the following aspects:

- quality of the raw materials, operation of reduction furnaces, ladle metallurgy, ingotting, crushing, grading and quality control.

It also describes the various types of nodulizers comparing them and showing their advantages and disadvantages.

Lastly, it describes the most used processes for introducing the nodulizing alloys in the base iron.

RAW MATERIALS

In order to produce a nodulizing alloy of good efficiency in obtaining ductile iron, it is of prime importance that the raw materials to be used in the manufacture of ferro-silicon, and that the elements used in the ladle metallurgy have a defined chemical composition, within the tolerance standards specified for each of these materials.

In defining the specifications of the raw materials, attempts were made to work with the lowest possible residues of other elements, and to achieve at the same time a consistency in the chemical analyses. Also, the size of the raw materials is very important, which must be adjusted to the reduction and fusion conditions of the production equipment.

Besides specifying the raw materials, it is necessary to have a precise and efficient Quality Control Plan, in order to detect and eliminate possible deviations from the established specifications.

The Quality Control has standardized the sampling methods, the frequencies and the types of tests to be carried out, for which laboratories for chemical analyses by instruments and wet method as well as for physical tests must be available.

There is also a constant search for new suppliers and/or the production of raw materials by the company itself which may improve and guarantee the quality of the products.

The specifications and the control plans of some of the raw materials are given as follows:

a) Quartz

- Chemical composition

SiO_2	minimum	99.5
Fe_2O_3	maximum	0.60%
Al_2O_3	maximum	0.20%
P_2O_3	maximum	0.05%
CaO	maximum	0.05%
MgO	maximum	0.05%

- Size
  about 75 mm

- Control Plan
  Monthly chemical analyses of various suppliers, representing 80% of the total quantities received (CaO; Al2; Fe2O3; SiO2 and loss through calcination).

  Thermostability tests for each of the samples to be analyzed chemically.

  Visual exam of each lot received.

b) Charcoal

At present two types of charcoal are used, that is, eucalyptus charcoal and "cerrado"* charcoal, with the tendency to use eucalyptys charcoal exclusively.

- Chemical Composition

  Fixed Carbon - minimum 70%
  Ashes - maximum 3%

- Size
  smaller than 12 mm - maximum 15%

- Control Plan

  Daily chemical analysis of quantities received (moisture, volatile materials, ashes and fixed carbon).

  Daily chemical analysis of the carbon used in the furnaces (moisture, volatile materials, ashes and fixed carbon).

  Montly chemical analysis of the ashes for each type of carbon used (CaO; Al2O3;Fe2O3; MgO; P2O5).

  Daily size test of the lot received. Daily size test of the carbon used in the furnaces, and determination of the apparent weight. Visual exam of each lot received.

c) Hematite

- Chemical composition

Fe	minimum	68.5%
Al_2O_3	maximum	0.80%
SiO_2	maximum	1.2%
P	maximum	0.02%
Mn	maximum	0.09%

- Size
  about 25 mm

- Control Plan

  Chemical analysis of each lot of 50 tons received (SiO2; CaO, Al2O3; P2O5; MgO; loss through calcination).

  Visual inspection of each lot received.

* It refers to a typical vegetation of certain regions in Brazil.

d) Metallic Magnesium

- Control Plan

  Chemical analysis of each lot received (Al)

e) Steel scrap

- Chemical composition

  Carbon steel

- State of materials delivered

  Scrap with dimensions suitable for use in the ladles. Free of oxidation, grease, oil, etc.

- Control Plan

  Visual inspection of each lot received.

f) Rare Earth Minerals

- Control Plan

  Chemical analysis of each lot received.

PRODUCTION OF BASE METAL IN REDUCTION FURNACES

Electric power received in the main substa~~n~~tion at 138 KV is converted into 13.2 KW through a 20/24 MVA transformer.

The transformer is equipped with taps for underload correction of oscillations in the system.

The base metal for manufacturing of Fe Si Ca Mg RE alloys is a Fe Si RE produced in furnaces of 6 MVA nominal.

Principal features

a) Three-phase Transformer

- Nominal rating : 6 MVA
- Primary feed : 13.2 KV
- Tap relations :

	Y/Δ		Δ/Δ	
	V	A	V	A
1	75.2	26604	130.2	26604
2	69.8	28630	121.0	28630
3	65.2	30652	113.0	30652
4	61.2	32683	106.0	32683
5	57.7	34600	100.0	34600
6	54.5	34600	94.5	34600
7	51.7	34600	89.5	34600
8	49.7	34600	85.2	34600

Charging of taps without any load

b) Furnace characteristics

- Diameter of electrodes : 750 mm
- Distance between centers of electrodes : 1850 mm
- Diameter of crucible : 3860 mm
- Height of crucible : 1820 mm
- 03 running holes
- Manual system for moving electrodes
- Rotating furnace

c) Operation of furnace

- Power : 5.4 - 5.5 MW
- Secondary voltage : 121 V
- Secondary current : 32 KA
- Cos φ : 0.82
- Utilization factor : 99.2%
- Intermitent pouring into ladles
- Number of runs per day : 9

d) Preparation of raw materials in the plant

- Quartz : washed and sieved
- Carbon : sieved

e) average recoveries

- Silicon : 95%
- Rare earths : 80%

ERRATA

1) Page 365- PRODUCTION OF BA

- First Column - 2nd para

  " The transformer is eq

  correction of the 138

- Principal features - F

  Please read: - 03 tap

  - Manual

LADLE METALLURGY

The base alloy, composed of iron, Silicon and Rare Earths, comes from the reduction furnaces. The final composition of the alloy is obtained in the ladles, by adding calcium, magnesium and steeel scrap to the primary alloy. In the process, two ladles with aluminum silicon lining are used, as follows:

- Receiving ladle with a capacity of 2.5 tons, which receives the Ferro-Silicon Rare Earths from the reduction furnace.
- Treatment ladle with a capacity of 3.5 tons, in which the additions are made.

The sequence of the process is the following:

- Remove slags from the Ferro-Silicon Rare Earths;
- Weigh;
- Place part of the magnesium in the treatment ladle (pre-heated), shortlly before transferring the Ferro-Silicon Rare Earths.
- Transfer part of the Ferro-Silicon Rare Earths to the treatment ladle;
- Add all the calcium;
- Add the rest of the magnesium;
- Add the rest of the Ferro-Silicon Rare Earths;
- Add the steel scrap;
- Homogeneize bath;
- Remove slag formed;
- Ingot

  During the introduction of the magnesium, the calcium and on ingotting, a neutral atmosphere is maintained through argon blowing in order to minimize the formation of oxides.
  Although it increases the process costs, the use of the argon is feasible due to the low costs of the raw materials.

The average time for the process is 35 minutes.

INGOTTING

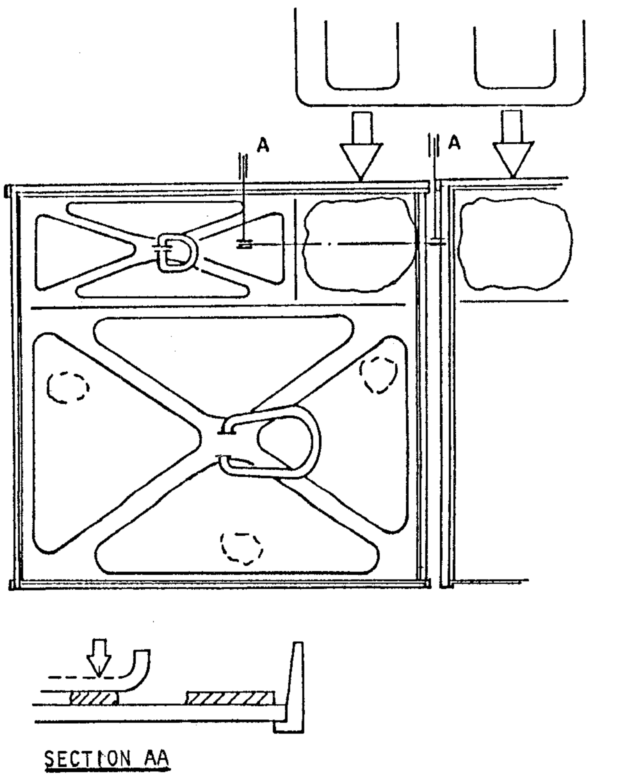
In order to guarantee the physical chemical properties of the alloy, the metal is poured in ingot molds with a cast iron cover. (Fig.01).

The bottom, sides and covers are cleaned after each run. Protectors of the same alloy are used to protect the ingot mold bottom. The position of the bottom is changed periodically to avoid excessive wear in the same area.

The ingots are broken and removed manually and samples are taken for chemical analysis, after which the material continues its production flow in crushing and grading areas.

Ingotting Features

- Ingot thickness : 30 to 50 mm
- Average weight of ingot: 450 kg
- Temperature of ingot moulds: 100 to 120°C
- Ingotting time : 30 ± 5 seconds/ingot
- Time between end of the ingotting and removal of the ingots: 10 ± 2 minutes.



Ingot Molds

Figure No 1

CRUSHING AND GRADING

The different process by which the nodulizing alloys are added demand greater efficiency of sizes which range from 0.59 to 50.80 mm.

The ingots are broken manually, with the objective of obtaining maximum sizes of 40 to 50 mm. If necessary, a further crushing is made with two jaw crushers, with typical openings of 31.60 mm in the primary and 25.40 mm in the secondary.

The sieve in vibrating screens with 2 or 3 decks after the crushing sequence above referred to, generates a typical size distribution, as follows:

Smallest 0.84 mm :	2.6%
0.84 - 6.35 mm :	8.0%
6.35 - 12.70 mm :	6.7%
12.70 - 19.10 mm :	3.3%
19.10 - 25.40 mm :	18.9%
25.40 - 31.60 mm :	18.7%
31.60 - 38.10 mm :	34.1%
Largest 38.10 mm :	7.7%

The material in its final form is packed in:

- plastic bags 50 Kg
- big bags 300, 500 and 1000 Kg
- steel drums 100 and 200 lts(200 to 300 Kg)
- others, by request

QUALITY CONTROL

1. Raw Materials

   All the raw materials are inspected before entering the plant, and samples are taken for size and chemical tests to check the elements listed in the specifications.

2. Pouring in the reduction furnace

   During the pouring operation samples are taken of each run for the purpose of determining Si contents of FeSiRE, for correcting the product still in the liquid state in the ladle.

3. Ingotting

   After solidification of the ingots, when these will be broken manually, samples are taken of each run to carry out the chemical analyses of the elements in the specifications.

4. Crushing and grading

   The sampling in the crushing and grading line has the objective of carrying out the final size tests and chemical analyses of the material already in the packing department.

   The frequency of the sampling is one sample for every 1000 Kg of product already processed.

   Many times, the assembly of the size curve specified by the client consists of the association of various size ranges processed during crushing and grading. In this case, new samples for size tests are needed.

5. Packing

   The various types of packings are inspected before and after introducing the materials in them.

   During this inspection, a check is made to assure that the containers are within the established norms (resistance, identification, waterproofing, , checking of seals after packing is completed, etc.)

6. Shipment

   The Quality Control follows up the loading of the vehicles which will transport the material, being sure that the containers are not damaged, and that their loading in the transporting vehicle will be done in such a way as to minimize eventual damage which may occur during transportation.

   Italmagnesio has a team of engineers specialized in transportation, who accompany the material until its final destination, supervising:

   a) Shipment in trains
   b) Transference from trains to ships
   c) Inspections in the ship holds
   d) Loading of the materials in the ship holds
   e) Follow-up of the unloading of the ships and the transportation to the final warehouses.

TYPES OF NODULIZERS AND COMPARISON OF TYPES

The nodulizing alloy is used in the manufacturing of ductile iron.

The nodulization can be obtained by adding pure magnesium, nickel-magnesium alloys, copper-magnesium alloys, magnesium impregnated coke, and ferro--silicon-magnesium-calcium-rare earths base alloys. The latter is the most used alloy in the world today.

The iron obtained through the addition of pure magnesium would be the most economical process, but today it is limited practically to the manufacture of centrifugal pipes, in which, due to a rapid cooling (pouring in chills) and a uniform geometric shape, a good nodulization can be obtained.

Up to this moment no post-inoculant has been found which would allow for the use of pure magnesium in other types of parts of asymetric shape and cast in sand, with good results.

The nickel magnesium alloy has a very good characteristic which is that of having a density very close to that of the base metal, and therefore a high magnesium recovery rate of up to 80% can be obtained. Nonetheles, it has as a disadvantage a very high cost because it contains nickel, and also the presence of this metal, which is not always wanted due to its pearlitizing effect.

Coppermagnesium alloys have characteristics similar to those of nickel-magnesium alloys.

Magnesium impregnated coke has an advantage when compared to pure magnesium. Because it is retained in the porosity of coke, it releases very slowly, giving a better magnesium recovery than when added pure, but on the other hand because the density is very low, the use of special techniques is necessary to prevent from floating.

FeSiCaMgRE alloys are the most used ones because they have shown the best characteristics for any type of ductile iron.

The effects of the elements which constitute the FeSiCaMgRE alloy are the following:

- Iron : gives the alloy greater density.
- Silicon : dissolues the magnesium when this element is introduced in the manufacture of the alloy; besides it is also a deoxidant, graffitizer and ferritizer.
- Magnesium : deoxidant, desulfurizer and nodulizer.
- Calcium : deoxidant, desulfurizer and graffitizer, besides increasing the speed of dissolution of the alloy when this is introduced in the base metal.
- Rare Earths: deoxidant, desulfurizer, graffitizer besides reducing the effects of the inhibitor elements.
- Aluminum : this element is always present in the alloys, being a deoxidant and a graffitizer, its presence however should be limited in the alloy, because when in excess it causes pin-holes ..

The percentages of the elements which constitute the alloy are variable and depend very much on the part to be manufactured.

The three types most commonly used are:

- Magnesium from 5 to 7%
- Magnesium from 8 to 10%
- Magnesium from 11 to 13%

The recoveries of magnesium of this alloy, until a few years ago, were not very high, between 30 and 40%, depending on the sulfur content in the base metal. In recent years, these recoveries have improved considerably, due to three principal factors:

- Percentage of the elements contained in the alloy
- Purity of the alloy
- Technology for introducing the alloy in the base metal

For these reasons, in the majority of the cases the Si, Mg, Ca and RE elements are almost always present in this type of nodulizers.

Calcium, which has a greater affinity to sulphur than magnesium, leaves the latter more free to act as a nodulizing agent, at the same time increasing the dissolution speed of the alloy, besides having a graffitizing action which provides more graphite to be nodulized.

Rare Earths are normally composed of 50% of cerium, 20% of lantanium and 30% of other elements such as praseodymium, neodymium, etc.

The cerium is the most efficient element in the Rare Earths to obtain the spheroidization of the nodules; however, other elements such as lantanium, praeseodymium, neodymium have a strong graffitizing effect (4).

The Rare Earths increase the nodule count per surface unit (4). The higher the nodule count, the better the characteristics of the ductile iron with whatever type of matrix desired. The characteristics are understood to be:

- Percentual elongation
- Tensile Strength
- Pouring limit
- Less formation of cementite

It could be said that cementite is nonexistent when the nodule count is high. The great advantage of this alloy, comparing to the nodulizing action of pure magnesium, is to have a simultaneous action of all the elements of the composition, so that the shape, the size and the quantity of nodules come very close to being ideal. In this manner, percentual elongations which reach 25% in ferritic ductile iron and up to 6% in pearlitic ductile iron are obtained. Another advantage is to be able to have the solidification point close to the euthetic, which at the time of solidification of the alloy in the mould provides finer crystals, and consequently a more compact and homogeneous alloy with little segregation. The temperature can also be increased at the beginning of the dissolution, giving more time for the formation of the metal column.

The modern technologies for the manufacturing and introduction of these alloys in the base metal are, to mention a few: TUNDISH COVER SYSTEM, IN MOLD SYSTEM, FLOW-THROUGH SYSTEM. When the size of the alloys is well adapted to the quantity of metal to be treated and to the shape of the ladle, magnesium recoveries of up to 80% are obtainable, which compares well with the recovery of the magnesium introduced in the form of nickel-magnesium alloy.

To obtain a good ductile iron and a good efficiency of the alloy, the latter should meet the following specifications:

- High purity
- Greatest possible density
- Chemical composition oriented to the parts to be manufactured
- Size compatible with the quantity of material to be treated.

When the alloy meets the characteristics mentioned above, a smaller quantity of it can be used, which allows for a lower production cost, besides introducing smaller quantities of silicon in the base iron. It also allows for the use of more returns and makes it possible to add a larger quantity of inoculants after the nodulization treatment, which no doubt will improve the physical and mechanical characteristics.

As we know, the post-inoculants regulate the chill depth, increase the number of euthetic cells and in certain cases eliminate the formation of dentrites of austenitic origin.

The purity of the alloy is very important because it permits only a lower formation of slag. We emphasize that when this nodulizer is introduced directly in the mold (In Mold System): any impurities will certainly appear in the part being manufactured.

The chemical analysis is very important: it not only influences the characteristics of the part, but also avoids violent reactions which, as we know, are polluting and, in certain treatments which are used throughout the world such as the Sandwich System, cause reactions of materials obviously dangerous.

The elements composing the alloy must under no circumstances be in the form of oxides or sulfites; they should be found in free state or combined adequately with other elements desired in the alloy.

Page 369- TYPES OF NODULIZERS AND COMPARISON O

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TECHNIQUES FOR APPLICATION OF THE NODULIZING ALLOYS

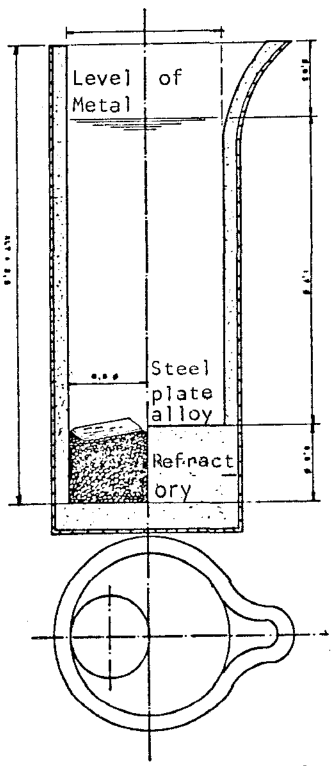
Sandwich System

It is the most widely used system for introducing the nodulizing alloy in the base iron. It is a cylindrical ladle the bottom of which has a lowered area where the alloy is deposited (fig. 02).

The relation between the inside diameter and the height of the ladle is approximately 1:2.5; this intense relation allows for a very high ferrostatic pressure.

To minimize the floatation of the alloy, several systems are used, the most common being to cover the alloy with steel sheets with weights varying between 1.0 to 1.5 times the weight of the alloy.

The loading of the ladle should be very quick to obtain in the least possible time a ferrostatic pressure which allows for a longer retention of the vapor in the metal and consequently a higher magnesium recovery.

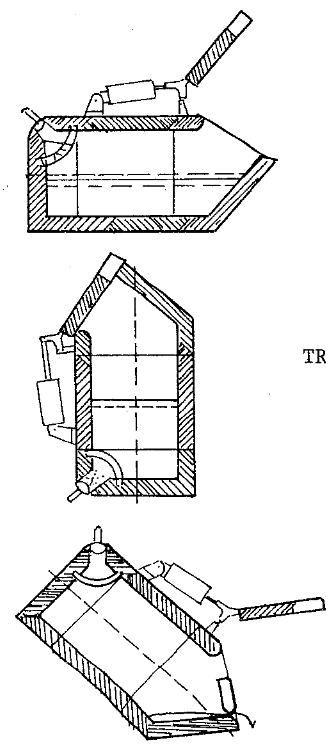


Sandwich System

Figure No 2

Fisher System

For pure magnesium a type of ladle is used which is very much like the Bessemer Converter, which has a false bottom where the magnesium is deposited (fig. 03). The metal is poured into this ladle, which is placed in a horizontal position so that the base metal will not enter in contact with the magnesium that is in the bottom. When all the metal has been poured this ladle is immediately placed in a vertical position so that, as in the previous case, in a very short period of time a ferrostatic column is produced which has the same effect as in the Sandwich System.



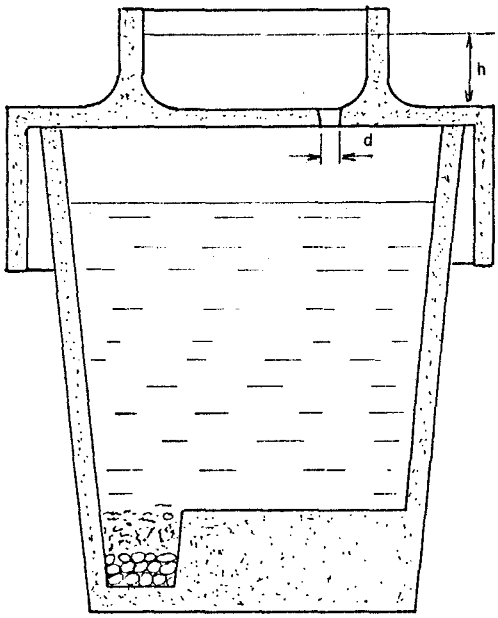
Fisher System (1)

Figure No 3

The quantity of alloys used in this type of ladles is very low, reaching 1% and leaving high residual values of magnesium in the parts. In this type of ladle, the great advantage is also a smaller loss of temperature through radiation. Therefore, the base metal can be poured from the furnace to the ladle at lower temperatures, which provides a smaller use of energy and a lower pressure of escape of magnesium.

Another great advantage in the use of this ladle is that it generates very low levels of polution which is one of the problems that exist in ductile iron foundries. The alloys mostly used in this type of ladle are low magnesium content alloys (5 to 7%) and 1% of cerium. We note that in this type of ladle we have obtained 80% of magnesium recovery, keeping the magnesium between 5.0 to 5.5% in the alloy. We are about to try alloys with a lower magnesium content (3.0 to 3.5%) and 1.5 of rare earths. The objective of such experiment is, besides attempting to increase the recoveries of magnesium, to obtain the least violent possible reactions.

We take the opportunity to point out once again the importance of the purity of the alloy, specially in this system, to obtain the greatest number of treatments, thus minimizing the cleaning of the ladle.



Tundish Cover System

Figure No 5

Flow-Through System

The treatment of the base metal is made during the passage through a system where the alloy is placed. This system reduces costs and allows for a very low pollution in the environment (fig. 06).

The reaction chamber differs from the In Mold System because it stays outside the mold and within the system indicated above. Despite having a magnesium recovery lower than that of the In Mold System, the following advantages are provided:

- Lower risk of slag entering the cast parts.
- Possibility of making post-inoculations.

When compared to the Sandwich System, it has the advantage of a low slag formation, also due to the fact that the system dispenses the use of steel sheets, minimizing the operation of removing them.

With this system we do not need a treatment ladle. The box containing the reaction chamber is placed below the furnace spout and the cast iron treated in the system passes directly to the transfer ladle.

The system has already been improved so that it allows for 50 to 60 treatments, nearly 60 tons. with the same box.

The reaction chamber shape is not so sensitive as in the In Mold System, it being possible to cast a wider range of parts with different dimensions with the same system.

The alloy which we find most appropriate for this type of system is a FeSiCaMgRE with the following chemical analysis:

Mg	=	3.0%
RE	=	1.5%
Ca	=	1.0%
Al	=	1.0%

This type of alloy, although already mentioned in the Tundish Cover system, has reactions that are not very violent when added to the base metal, and therefore not very polluent.

The magnesium recovery of the alloy, when the purity and the size are adequate, reaches 60%.

Immersion System

Another system for the introduction of the metallic magnesium or the magnesium ferro-silicon base alloy is the immersion system where a bell is used which is introduced quickly until the bottom of the ladle when this is full so that the dissolution will start when the nodulizer has reached the bottom.

These last two systems have the disadvantage of not being able to be used for small quantities of materials. These processes take longer than the Sandwich Sistem and are normally used in factories producing centrifugal pipes.

In Mold System

The In Mold System consists in placing the rare earth magnesium ferro-silicon base alloys in the mold (fig. 04).

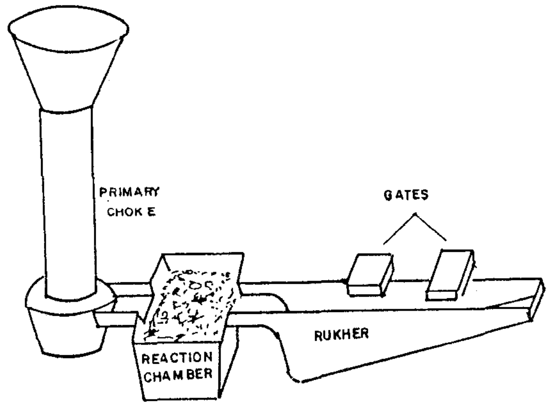
This system has several advantages:

- The parts cast in this system present characteristics superior even to forged steel.
- The quantity of alloy used is about 0.9%, therefore a very low consumption of alloy.
- There is no fading problem.
- The quantities remaining in the ladle continue to have the same analysis than that of the base iron.
- The channels before the reaction chamber are easily broken as this is a gray cast iron.

As the calculation of the reaction chamber of the finger gates in the feeding system is complicated, this system is only justified in the case of large productions in series, and for this reason limited to the automotive industry.

We take the opportunity to say something more about the effect of the calcium action in the dissolution of the alloy in the cast iron.

Normally, the calcium contained in the In Mold alloys is limited in values ranging from 0.30 to 0.70%. It can easily be seen that, when this element is above the maximum value, the part opposite to that of the treated metal becomes highly nodulized, and that the part filled in last of all practically does not present any nodulization With values under the minimum stated the opposite effect is observed.



In Mold System

Figure No 4

Tundish Cover System

It is the system most used at the present time. The ladle has a lowered area where the alloy is deposited, in the same manner as in the Sandwich System. The alloy is covered with steel sheets. The ladle is covered with another ladle having a hole in its bottom, the diameter of which can be calculated through the following formula: (1)

$$R = C\sqrt{2g} \quad x\sqrt{h} \quad x \frac{\pi d^2}{4} \text{ (cm}^3\text{/seg), where:}^1$$

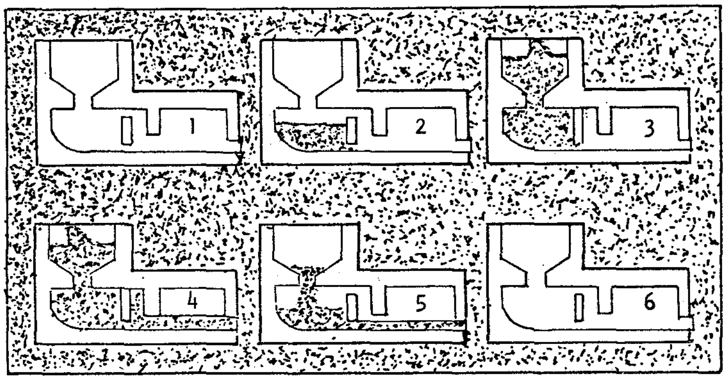
C = friction coefficient = 0.7 (approximately)
g = acceleration of gravity (cm/s^2)
h = see figure (cm) (fig.05).
d = see figure (cm) (fig.05).

The metal is poured in this intermediate ladle which should be kept full at all times.

The base metal flows through this hole, enters the lower ladle and, shortly after, the reaction begins.

As the intermediate ladle closes almost completely the lower ladle, the first magnesium vapors react with the oxigen in the air contained in the ladle, neutralizing it. As the metal goes up, the gases held in the intermediate ladle oppose themselves to the outlet of the magnesium vapor, and this contrary pressure increases rapidly as the space diminishes due to the expansion of the imprisoned gases and also due to the increase in temperature. Consequently, by increasing the retention of the magnesium vapors in the metal, the recovery of this metal is also increased.

We believe that in the near future this system will be improved and will be able to be used in almost all types of foundries.



Flow-Through System
Figure No 6

PRESSURE OPERATED POURING UNIT - TYPE GED

This new system consists in an induction furnace (see fig. No 07) which works as a large holding and has the following advantages:

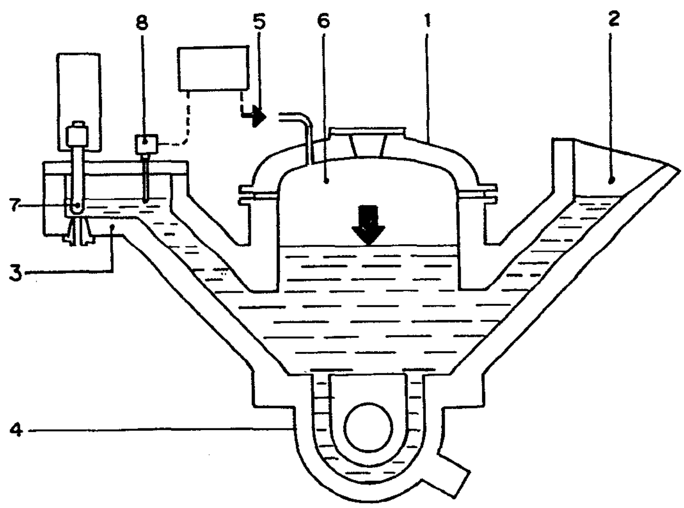
a) Pourings of metal without slag controlled at constant temperature

b) Shorter pouring periods.

c) Pourings directly into the mold, with preset loading time and metal quantities, due to a valve and a rod with a cover on the pouring head (similar to an ingot ladle used in steel industry).

d) Constant flow of liquid metal because the furnace is pressurized and the pressure is automatically controlled (in the case of ductile iron the crucible is pressurized with nitrogen).

e) The pressurization increases the fading time. The combined use of Tundish Cover System and this new George Fisher System is giving good results. The nodulizing alloy recommended should have a maximum of 5,5% magnesium.



Functional diagram of pressure operated pouring unit

1 pouring vessel
2 filling funnel
3 pouring head
4 inductor
5 pressure feed
6 vessel chamber
7 stopper
8 float

Pressure Operated Pouring Unit - Type GED (7)
Figure No 7

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