The PQ-DIT process. Improving Ductile Iron production using ultra-low additions of magnesium.

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Introduction:  
Almost every foundry that uses batch type treatments to produce ductile iron adds a constant amount of magnesium to each ladle. The amount of the addition, usually consisting of a ferro silicon magnesium alloy, has been found by trial and error to suit the specific type of production that exists in the foundry. A typical addition is 1.5%. If the amount of residual magnesium is above a certain minimum level and other critical elements are within acceptable limits then the dissolved carbon will precipitate as spheroidal shaped graphite. A foundry with heavy castings with large modulus might aim at a minimum magnesium level of 0.055%. For another foundry with thin-walled castings and short times between treatment and pouring a minimum level of 0.035 might be sufficient. These levels have to be high enough to take care of "worst case" metallurgical variations in the base iron (oxygen, sulphur, nitrogen, nucleation, etc.) and in Mg-yield. Although these levels do produce acceptable ductile iron, most of the time they are too high and on average too much magnesium is used. The result is unnecessarily high costs, problems with dross-inclusions, micro shrinkages and reduced machinability.

Ideally, the magnesium addition should be varied depending on the metallurgical status of the base iron, i.e. total oxygen and sulphur, active carbon equivalent, nucleation potential, treatment temperature, etc. However, this has not been possible until now, as the foundries have lacked practical and adequate measurement systems. Chemical analysis with a spectrometer is simply not sufficient. It only quantifies the amount of each element but does not tell us anything about in what form the elements are present (e.g. oxides, sulphides, silicates) and even less about how the melt does nucleate during solidification. A magnesium content of 0.045% might consist of 0.015% dissolved magnesium, 0.01% as MgO, 0.020% as MgS and Mg-silicates and nitrates and give a 95% nodularity. However, on another occasion the same total magnesium level of 0.045% might result in only 50% nodularity due to a lower amount of dissolved magnesium. The reason might be higher levels than usual in the base iron of elements that tie up the magnesium such as oxygen, sulphur or nitrogen. This happens every now and then, but as it cannot be detected by spectrometer analysis, the foundry is forced to always use a high magnesium content sufficient for the worst case, e.g. 0.055% in order to be able to produce ductile iron with 95% nodularity. The purpose of the PQ-DIT process control system developed by NovaCast AB is to solve this problem and allow the foundry to use the minimum amount at all times.
The metallurgical status of the base iron can fluctuate considerably. The total oxygen level might vary from 50 – 100 ppm, nitrogen from 40 – 120 ppm, sulphur from 0.008 – 0.015 etc. The nucleation ability of the base iron which can be measured by thermal analysis will influence the final nodularity and also here the variation can be high. The illustration below show two cases which are realistic to happen in a foundry. Case 1 represents a situation where a magnesium level of only 0.025% would give a perfect ductile iron. In Case 2 a magnesium level of 0.05% is required in order to obtain the same nodularity.

![Graph showing nodularity vs. magnesium level](image)

| Mg yield % | 70 | 65 |
| Sulphur % | 0.010 | 0.015 |
| Oxygen ppm | 60 | 80 |
| Nitrogen ppm | 50 | 90 |
| Nucleation | High | Low |

If the foundry can not measure the status of the base iron they must always add enough magnesium to reach the 0.05% level. In reality each melt is an individual and the required magnesium level varies between these two extremes. The conclusion is that every foundry producing ductile iron with traditional methods are using too much magnesium.

**Prime Quality Ductile Iron Technology:**
PQ-DIT, is an abbreviation for Prime Quality Ductile Iron Technology. It is a new concept, developed by NovaCast, in order to produce high quality ductile iron with ultra-low magnesium additions. The technology is based on advanced thermal analysis of the base iron combined with chemical analysis. The system estimates total oxygen as well as oxygen activity in the melt, the active carbon equivalent, the nucleation status and several other important metallurgical parameters. Based on these data and the chemical composition, PQ-DIT evaluates the melt and if needed recommends additions of special conditioners to adjust the base iron before treatment.
When the base iron is within the acceptable process "window", PQ-DIT calculates the ideal amounts of magnesium and other additives to be used when treating the iron. The “recipe” produced is specific for each alloy and type of casting category. The PQ-DIT concept also includes consultancy assistance in selecting optimal alloys and in improving melting and treatment technology.

The starting point is a good base iron. In order to produce a high quality ductile iron the major requirements on the base iron are:

The active carbon equivalent (ACEL) must be selected to suit the casting to be made and the mould. The active carbon equivalent and the silicon content determine how much eutectic carbon can be used for reducing the contraction during solidification. The active carbon equivalent is measured with the PQ-DIT system.

The nucleation properties of the melt must be within acceptable limits. This can be ensured by controlling parameters such as the low eutectic temperature, the recalescence and specific graphite precipitation factors. By controlling these factors with PQ-DIT it is possible to optimise the amount and precipitation pattern for graphite and thereby reduce or even eliminate the use of feeders.

The total oxygen level must be within a specific range. Note that a measurement of the active oxygen only shows a small part of the oxygen in the melt. The important factor is the amount of total oxygen, because also oxides are reduced by magnesium. PQ-DIT estimates total oxygen and also oxygen activity.

The metallurgical “fingerprint”, i.e. important thermal parameters, measured by PQ-DIT, must be within accepted control limits. This is done by using an alloy profile diagram and a comparison with predetermined successful limits.

The chemical composition must of course also be within acceptable limits. It is especially important that the sulphur content is known with a high accuracy. Acceptable sulphur levels for the base iron are 0.008 to 0.018%.

**Testing and adjusting the base iron with PQ-DIT 1:**

When the base iron is melted and at the normal final holding temperature, a sample is poured using standard Quik-Cups. Two cups are used for each test – one standard cup with tellurium and one without.

The PQ-DIT system evaluates the iron by using the parameters mentioned above and suggests any adjustments needed for the base iron before the magnesium
treatment. That includes additions of alloying elements as well as correction of the oxygen content.

When the base iron is “conditioned”, the PQ-DIT system suggests the ideal additions of FeSiMg, cover material and Ce-MM to be added to the treatment ladle. This recipe can then be used for several ladles without the need to pour a new sample.

The variations in “active” magnesium mainly depend on:

- The FeSiMg-alloy, its composition and grain size
- The design of the alloy chamber
- The design of the treatment vessel
- Type and amount of cover material
- Treatment temperature
- Dwell time for the alloy in the alloy chamber (if hot) before treatment
- The total oxygen level in the base iron (See picture below)
- The sulphur level in the base iron
- The nitrogen level in the base iron
- The nucleation level in the base iron

The screen shows the result after testing a base iron. The OK sign tells the operator that the melt is ready for treatment.

The Mg-yield often varies between 55 and 70% from ladle to ladle due to variations in one or several of the mentioned factors unless special precautions are taken. The PQ-DIT technology includes know-how to reduce the variations. Variations in oxygen and sulphur also make a big difference. A base iron with a sulphur content of 0.01% and an oxygen content of 40 ppm might require a total addition of about 1.1% of an FeSiMg alloy. If sulphur and oxygen increase to 0.015% and 80 ppm, then an addition of about 1.35% would be needed in order to achieve the same nodularity. If there were a variation in other factors that influence the Mg-yield, causing the yield to drop 15%, then the need for FeSiMg alloy would increase from 1.35 to 1.60%.
The PQ-DIT process control system allows the foundry to control oxygen, sulphur and nucleation properties and by adjusting the additions of FeSiMg dynamically we can reduce the additions considerably as the example above demonstrates. If the other factors are also considered to increase the yield and the repetition accuracy then further reductions are possible. The PQ-DIT concept also includes technical advice about how to improve the yield factors, selecting alloys and inoculants, etc. The picture shows the screen with the recommended additions for the current melt.

**Verification of the final iron with PQ-DIT 2:**
The final treated iron can be verified by pouring two Quik-Cups and analysing the cooling curves with the PQ-DIT 2 system. One of the cups contains tellurium. The effect is that Te combines with the dissolved Mg in the iron forming magnesium telluride (MgTe). The iron in that cup solidifies therefore almost like a normal gray iron. By comparing thermal data between the two cooling curves it is possible to estimate the percentage of dissolved magnesium as well as the nodularity in the iron (see screen shot). Acceptable limits can be set in the alloy database for different alloys and/or specific castings. The PQ-DIT 2 system needs to be calibrated for each installation, as it is dependent on the process used.

The data from PQ-DIT are also used to fine-tune the whole treatment process. When installing the system the target for final Mg should be gradually reduced in order to find an optimal operating level.
Benefits:
The PQ-DIT system offers a reproducible way of producing ductile iron with very low levels of magnesium. The main benefits are:

- Less tendency for micro shrinkage defects
- Possibility to increase casting yield by reducing the need for feed metal
- Less tendency for dross and other inclusions
- Better machinability
- Increased process stability
- Reduced costs – the Pay-Off time is often less than 6 month.

The micros below shows an improved nodularity with the PQ-DIT treated iron although the magnesium level is reduced by 38%!

<table>
<thead>
<tr>
<th>Traditional treatment</th>
<th>With PQ-DIT and special Elkem alloy</th>
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<tbody>
<tr>
<td>Mg=0.042%</td>
<td>Mg=0.026%</td>
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Availability:
The PQ-DIT system consists of two parts. PQ-DIT 1, which is used to control and adjust the base iron and to recommended ideal additions for the magnesium treatment. This system should be installed at the melting deck or where the holding furnaces are.

PQ-DIT 2 is the verification system. It should be installed close to the pouring area as it is used to test and verify the final, treated iron.

The Prime Quality Ductile Iron Technology systems are supported by world-class specialists in foundry technology and metallurgy from NovaCast and Elkem.
The PQ-DIT process flow can be illustrated as follows:

1. Test Base Iron
2. Condition Base Iron
3. Optimal Ladle Additions
4. Mg treatment
5. Transfer to pouring ladle
6. Pouring
7. Test Final Iron

Chemical Analysis
Treatment Temp.
Casting Spec. Data