Analysis of the Graphite Shape in Cast Iron Obtaining by Inmold Process

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Abstract
The paper presents results of research on the effect of magnesium on the graphite shape in cast iron obtained by Inmold process. For testing Lamet® 5504 master alloy was used. The crystallization process of cast iron with various Mg concentrations was evaluated with use of the thermal and derivative analysis (TDA) method. The conditions were demonstrated for which a flake, vermicular and nodular graphite were obtained. Values of graphite “c” coefficient were analyzed. Results from it the significant differences between the assessment of the graphite on metallographic specimen and by the “c” coefficient. An effect of wall thickness of the casting on the graphite coefficient was indicated.

Keywords: Theory of crystallization, Inmold process, Graphite shape

1. Introduction
Cast iron modification processes aimed at changing the morphology of graphite have been known since 1940s. At present, many processes and their variations in both nodularising and vermicularising of cast iron are known [1-11]. In practice to nodularising process iron are currently used [6]:
- placing of master alloys using bells made of different materials,
- by pouring the reagents placed on the bottom of a ladle (Sandwich, Trigger or Tundish process),
- introducing Mg-Al rods into the sealed ladle (JPK method),
- making a pure magnesium in a converter ladle,
- making Mg into the ladle placed in the autoclave,
- making magnesium in the carrier gas stream through a porous stopper located at the bottom of the ladle (Osmose method),
- methods using special design of tapping runners (FLOTRET [2, 3] and IMCONOD methods),
- cored wire treatment (PE method),
- nodularising in a magnetic field (HTM method),
- Inmold process.
All of the above mentioned methods are designed to make nodular graphite. To the most commonly methods of vermicularising treatments (incomplete nodularising) include [6]:
- making liquid cast iron with rare earth elements,
- the introduction of magnesium as silico-magnesium master alloy (eg. by Inmold process),
- the treatment of cast iron with master alloys containing Mg and Ce and anti-nodulising elements such as Ca, Ti and Al,
- the treatment of cast iron with magnesium-zirconium master alloy.
An effect of a nodularising (or vermicularising) process and inoculation is obtaining the nodular (or vermicular) graphite. The shape of graphite in cast iron is described in the Polish Standard PN-EN ISO 945-1: 2008. It comprises six standards of the shape marked I – VI. There are shown in Figure 1.
According to the above mentioned standard a nomenclature of the graphite is as follows:

- I flake graphite,
- II rosette graphite,
- III vermicular (compacted) graphite,
- IV temper graphite,
- V slightly irregular nodular graphite,
- VI nodular graphite.

The given standards allow the assessment of graphite on the basis of metallographic tests.

In the world literature there is a lot of information on the possibility of assessing the shape of graphite [12-18]. There are many methods to assess the stereological features of graphite, allowing formulate its coefficient [19-23]. Our study enabled conclude the significant differences between the visual evaluation of graphite, and the numeric values obtained by analysis of the image. Therefore, the aim of the study was to evaluate the shape of graphite in cast iron with different Mg concentration obtained by Inmold technology. This method was chosen because of its high efficiency [9].

2. Research methodology

Initial cast iron was melted in an electric induction crucible furnace Elkon PI 30 of 30 kg capacity and 3.8 kHz frequency. As the charge a special pig iron with the chemical composition shown in Table 1 was used.

Table 1.
Chemical composition of special pig iron

<table>
<thead>
<tr>
<th></th>
<th>C</th>
<th>Si</th>
<th>Mn</th>
<th>P</th>
<th>S</th>
</tr>
</thead>
<tbody>
<tr>
<td>wt. %</td>
<td>4.44</td>
<td>0.69</td>
<td>0.025</td>
<td>0.046</td>
<td>0.009</td>
</tr>
</tbody>
</table>

The content of silicon and manganese was controlled by addition FeSi75 and FeMn75, respectively.

The chemical composition of cast iron was tested with using emission spectrometer with spark excitation SPECTROMAXx made by SPECTRO Analytical Instruments GmbH. The composition of the selected melts along with the eutectic carbon equivalent \( C_e \) and the degree of eutectic saturation \( S_c \) were given in Table 2.

Table 2.
The chemical composition of cast iron, its eutectic carbon equivalent \( C_e \) and the eutectic saturation ratio \( S_c \)

<table>
<thead>
<tr>
<th>Chemical composition, wt.%</th>
<th>( C_e )</th>
<th>( S_c )</th>
</tr>
</thead>
<tbody>
<tr>
<td>C</td>
<td>Si</td>
<td>Mn</td>
</tr>
<tr>
<td>-----</td>
<td>-----</td>
<td>-----</td>
</tr>
<tr>
<td>4.06</td>
<td>1.89</td>
<td>0.33</td>
</tr>
<tr>
<td>3.98</td>
<td>1.93</td>
<td>0.30</td>
</tr>
<tr>
<td>4.10</td>
<td>2.62</td>
<td>0.37</td>
</tr>
<tr>
<td>4.09</td>
<td>2.64</td>
<td>0.35</td>
</tr>
<tr>
<td>4.23</td>
<td>3.21</td>
<td>0.37</td>
</tr>
<tr>
<td>3.93</td>
<td>3.15</td>
<td>0.34</td>
</tr>
<tr>
<td>3.88</td>
<td>3.10</td>
<td>0.35</td>
</tr>
<tr>
<td>4.02</td>
<td>3.12</td>
<td>0.35</td>
</tr>
</tbody>
</table>

Eutectic equivalent of carbon was calculated according to the formula [5]:

\[
C_e = C_t + 0.30 \cdot Si - 0.027 \cdot Mn + 0.40 \cdot S + 0.36 \cdot P
\]  \( (1) \)

where \( C_t \) – the total carbon content in the cast iron, wt. %.

The eutectic saturation ratio was calculated as follows:

\[
S_c = \frac{C_e}{C_{sat}}
\]  \( (2) \)

where \( C_{sat} \) – the carbon content in the eutectic graphite calculated according to [5]:

\[
C_{sat} = 4.26 - 0.30 \cdot Si + 0.027 \cdot Mn - 0.40 \cdot S - 0.36 \cdot P
\]  \( (3) \)

The nodularising and vermicularising treatment of cast iron was done by an Inmold process. The scheme of the gating system components is shown in Figure 2.

Fig. 2. Schematic layout of elements in the mould to a nodularising and inoculation of cast iron by Inmold process
1 – downsprue, 2 – reaction chamber, 3 – mixing chamber,
4 – control chamber, 5 – test casting, 6 – flow-off

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Fig. 2. Schematic layout of elements in the mould to a nodularising and inoculation of cast iron by Inmold process
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4 – control chamber, 5 – test casting, 6 – flow-off
With cast iron of the temperature of about 1480°C the mould was poured through a sprue (1). A nodularising and inoculating master alloy Lamet® 5504 Elkem was placed in the spherical reaction chamber (2) of ø 85 mm diameter. The chemical composition of master alloy is shown in Table 3. Behind the reaction chamber there was a mixing chamber (3) which allowed the accurate dissolution of the master alloy and provide high yields of magnesium [24]. Behind mixing chamber there was a control chamber (4). In the thermal center of this control chamber, a PtRh10-Pt thermocouple was placed to record temperature changes during cast iron cooling and solidification. The thermocouple was connected with U/f converter through a compensation wire and then with a computer. This allowed, in turn, plotting the TDA curves. Then liquid cast iron filled cavity (5) in the shape of a stepped test casting.

<table>
<thead>
<tr>
<th>Table 3. Chemical composition of Lamet® 5504 master alloy</th>
<th>Chemical composition, wt. %</th>
</tr>
</thead>
<tbody>
<tr>
<td>Si</td>
<td>Mg</td>
</tr>
<tr>
<td>44±48</td>
<td>5±6</td>
</tr>
</tbody>
</table>

Metallographic examinations were carried out under a Nikon Eclipse MA200 optical microscope at a magnification of ×200. The shape of graphite precipitations was examined by means of a NIS-Elements BR image analysis program. To assess the shape of the graphite following formula was used [19]:

\[ c = \frac{4 \cdot \pi \cdot A}{L^2} \]  \hspace{1cm} (4)

where:
- \( A \) – graphite precipitation’s surface,
- \( L \) – graphite precipitation’s perimeter.

Generally the formula for graphite coefficient is known [20, 22]:

\[ c = \frac{O_k}{O_w} \]  \hspace{1cm} (5)

where:
- \( O_k \) – circumference of a circle with an area of a graphite precipitation,
- \( O_w \) – graphite precipitation’s perimeter,

and the ensuing ranges of values according to which the graphite is determined as nodular (0.80 ≤ \( c \) < 1.00), vermicular (0.66 ≤ \( c \) < 0.80) and flake (\( c < 0.66 \). Taking into account the equations (4) and (5) following values of “c” coefficient were obtained:
- nodular graphite: 0.64 ≤ \( c \) < 1.00,
- vermicular graphite: 0.43 ≤ \( c \) < 0.64,
- flake graphite: \( c < 0.43 \).

3. Results

Figure 3 shows the TDA curves plotted for a gray cast iron.

Since this is a hypereutectic cast iron, the crystallization starts from the primary graphite precipitate. The thermal effect coming from this process is not visible on the derivative curve due to the small amount of heat generated during the transformation \( \text{L} \rightarrow \text{C}_{\text{pt}} \) (liquid → graphite). The transformation of the liquid into the eutectic mixture of austenite and flake graphite is shown on the derivative curve as thermal effect described by DEFH points. Recalcescence of the temperature amounted to 7°C. The solidification of cast iron ended at 1088°C. For the other types of tested cast iron the crystallization process was similar, hence in Table 4 values of the temperature at the points describing TDA curves were shown.

<table>
<thead>
<tr>
<th>Table 4. Values of the temperature at the points describing TDA curves of tested cast iron</th>
</tr>
</thead>
<tbody>
<tr>
<td>Mg concentration, wt. %</td>
</tr>
<tr>
<td>------------------------</td>
</tr>
<tr>
<td>0</td>
</tr>
<tr>
<td>0.0044</td>
</tr>
<tr>
<td>0.0089</td>
</tr>
<tr>
<td>0.018</td>
</tr>
<tr>
<td>0.028</td>
</tr>
<tr>
<td>0.039</td>
</tr>
<tr>
<td>0.048</td>
</tr>
<tr>
<td>0.067</td>
</tr>
<tr>
<td>0.090</td>
</tr>
</tbody>
</table>

It should be noted that increasing the crystallization temperature by increasing the concentration of Mg is caused mainly by the increase in concentration of Si introduced together with Mg in the master alloy.

Figures 4-6 (a, b) show the shape of graphite in castings with a wall thickness of 3 and 24 mm made of gray cast iron (Fig. 5), vermicular cast iron (Fig. 6) and nodular (Fig. 8).
Fig. 4 (a, b). The shape of graphite in gray cast iron castings with a wall thickness of 3 (a) and 24 mm (b) (Mg = 0.028%)

Fig. 5 (a, b). The shape of graphite in vermicular cast iron castings with a wall thickness of 3 (a) and 24 mm (b) (Mg = 0.028%)

Fig. 6 (a, b). The shape of graphite in nodular cast iron castings with a wall thickness of 3 (a) and 24 mm (b) (Mg = 0.048%)

From Fig. 4-6 shows the greater refinement of graphite in thin walled casting (3 mm) in comparison to the thick walled (24 mm).

Values of the graphite coefficient “c” as a function of Mg concentration for castings with a wall thickness of 3 and 24 mm is shown in Table 4 and graphically in Fig. 7.

Table 5.
Values of the graphite coefficient “c” as a function of Mg concentration for castings with a wall thickness of 3 and 24 mm

<table>
<thead>
<tr>
<th>Mg concentration, wt. %</th>
<th>3 mm</th>
<th>24 mm</th>
</tr>
</thead>
<tbody>
<tr>
<td>0.0044</td>
<td>0.186</td>
<td>0.149</td>
</tr>
<tr>
<td>0.0089</td>
<td>0.19</td>
<td>0.174</td>
</tr>
<tr>
<td>0.018</td>
<td>0.51</td>
<td>0.466</td>
</tr>
<tr>
<td>0.028</td>
<td>0.562</td>
<td>0.479</td>
</tr>
<tr>
<td>0.039</td>
<td>0.627</td>
<td>0.599</td>
</tr>
<tr>
<td>0.048</td>
<td>0.779</td>
<td>0.712</td>
</tr>
<tr>
<td>0.067</td>
<td>0.727</td>
<td>0.741</td>
</tr>
<tr>
<td>0.090</td>
<td>0.712</td>
<td>0.711</td>
</tr>
</tbody>
</table>

R² = 0.9579

R² = 0.9691

![Graph showing values of “c” coefficient vs magnesium concentration in castings with a wall thickness of 3 and 24 mm made of cast iron obtained by Inmold process](image)

From Fig. 7 follows a slightly higher value of the “c” coefficient in castings with a wall thickness of 3 mm in comparison to castings with a wall thickness of 24 mm. It results from greater refinement of graphite in the thin walled castings. From the data presented in Fig. 7 also shows that vermicular cast iron was obtained for Mg concentration at about 0.02%, while the nodular cast iron for Mg = 0.04%. In cast iron containing 0.067% Mg first precipitations of “chunky” graphite were observed. They are shown in Figure 8.

Fig. 7. Values of “c” coefficient vs magnesium concentration in castings with a wall thickness of 3 and 24 mm made of cast iron obtained by Inmold process

Fig. 8 (a, b). An example of “chunky” graphite in castings with a wall thickness of 3 (a) and 24 mm (b) in cast iron with Mg = 0.067%

Graphite coefficient “c” in standards from PN-EN ISO 945-1:2008 shown in Fig. 1 was evaluated. The results are shown in Table 6.
Table 6. Values of “c” coefficient for standards from PN-EN ISO 945-1:2008

<table>
<thead>
<tr>
<th>No. of standard according to PN-EN ISO 945-1:2008</th>
<th>Value range of “c” coefficient</th>
<th>Average value of “c” coefficient</th>
</tr>
</thead>
<tbody>
<tr>
<td>I</td>
<td>0.037 – 0.649</td>
<td>0.222</td>
</tr>
<tr>
<td>II</td>
<td>0.047 – 0.642</td>
<td>0.222</td>
</tr>
<tr>
<td>III</td>
<td>0.135 – 0.665</td>
<td>0.453</td>
</tr>
<tr>
<td>IV</td>
<td>0.164 – 0.909</td>
<td>0.527</td>
</tr>
<tr>
<td>V</td>
<td>0.321 – 0.878</td>
<td>0.640</td>
</tr>
<tr>
<td>VI</td>
<td>0.853 – 0.990</td>
<td>0.950</td>
</tr>
</tbody>
</table>

From the data presented in Table 6 follows a large range of values of “c” coefficient for the flake graphite (standard I), vermicular (III) and slightly irregular nodular (V). In each of them there are precipitations with the “c” coefficient corresponding to the flake and nodular graphite.

Examples of various graphite precipitations in examined cast iron and its “c” coefficient are shown in Figure 9 (a-i).

![Fig. 9 (a-i). Examples of various precipitations of graphite in the examined cast iron and its “c” coefficient](image)

It follows from this that the graphite evaluated as vermicular on the metallographic specimen is classified by values as flake (eg. Fig. 9b), while similar to the nodular (Fig. 9e) - as vermicular.

The relationship between the type of graphite and Mg concentration in the casting with a walls thickness of 3 and 24 mm shown in Figure 10 (a, b).

It follows from this that the nodular graphite has performed up to a concentration of about 0.04% Mg, and the first nodular precipitations was observed at a concentration of about 0.01% Mg in the whole range of the wall thickness. In the thin walled castings, graphite classified by “c” coefficient as vermicular has already occured at a concentration of Mg = 0.0044%. The first "chunky" graphite were observed at a concentration of about 0.05% Mg, wherein its fraction increased more in the thin walled castings.

4. Summary

The data presented in the paper indicate significant differences in the classification of graphite based on the evaluation on metallographic specimen and according to "c" coefficient. In many cases, graphics evaluated visually as vermicular is characterized according to the "c" coefficient as flake graphite. Sometimes graphite having a shape similar to a nodular has a "c" coefficient characteristic for vermicular graphite.

The results showed the possibility of using the Inmold process to nodular as well as vermicular cast iron production. In both
cases there were graphite precipitations classified by “c” coefficient as a flake and vermicular. Considering the average value of coefficient vermicular cast iron was obtained at Mg concentration of about 0.018%, and nodular – of about 0.04%. It has been found that in thin walled castings there is a graphite having a higher coefficient, particularly in gray cast iron and vermicular; this involves a greater its dispersion.

References


