Original Article

Effect of holding time, thickness and heat treatment on microstructure and mechanical properties of compacted graphite cast iron

Hassan Megahed\textsuperscript{a}, Emad El-Kashif\textsuperscript{a}, Ahmed Y. Shash\textsuperscript{a,b,*}, Mahmoud A. Essam\textsuperscript{c}

\textsuperscript{a} Mechanical Design and Production Dept., Faculty of Engineering, Cairo University, Cairo, Egypt
\textsuperscript{b} Faculty of Engineering and Materials Science, German University in Cairo, Cairo, Egypt
\textsuperscript{c} Higher Technological Institute (HTI), 10\textsuperscript{th} of Ramadan City, Egypt

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ABSTRACT

The effect of holding time, thickness and annealing heat treatment on the microstructure and some mechanical properties of compacted graphite iron (CGI) are studied. Samples of CGI are produced in Helwan factory for casting by using GGC 70 as base metal in a medium frequency induction furnace. The mechanical properties (tensile strength, and hardness) of the as – cast and after heat treatment samples are determined and the microstructure of the samples is examined using optical microscope. The results show that the mechanical properties and microstructure of CGI depend on holding time, thickness and annealing heat treatment; it is found that increasing the holding time from 10 min to 17 min results in lowering the Mg content from 0.031\% to 0.021\% and as a result lower nodularity was obtained. Lowering the thickness from 20 mm to 5 mm increases the tendency of dendritic structure as a result of increasing the cooling rate. The annealed samples with mainly ferritic matrix gave the lowest tensile strength and hardness value compared with the as-cast conditions.

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1. Introduction

Compacted or vermicular graphite cast irons (CGI) have been produced since the beginning of spheroidal graphite cast irons in the late 1940s [1]. Compacted graphite cast iron was produced by accident due to the breakdown of process control during production of ductile cast iron. The importance of CGI moves toward stronger, lighter materials, applications have been found for the unique mechanical properties offered by the compacted form of graphic irons. High mechanical properties with good thermal conductivity and damping capacity gave many applications for CGI such as diesel engine blocks, exhaust manifolds and ingot molds [2].

It is well known that the mechanical properties [3,4] and microstructure [5] of spheroidal graphite cast irons (SGI), also known as ductile or nodular cast irons, depend on the casting size [6,7]. In particular, the larger the casting size, the lower the nodule count and nodularity and the larger the nodule size [7]. This results in lower mechanical properties. Different graphite

\* Corresponding author.
E-mail: ahmed.shash@cu.edu.eg (A.Y. Shash).

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morphologies can thus be obtained by varying the casting wall thickness, especially in the case of heavy section castings [6,8]. By also considering the work of Kalibom et al. [9], it is clear that many factors affect CGI degeneration: (i) cleanliness of the initial charge and bath handling; (ii) pouring temperature; (iii) inoculation; (iv) magnesium treatment; (v) solidification time and sequence.

The creation of high caliber of CGI requires precise determination of materials and close metallurgical control to get uniform and homogeneous compacted graphite structures, this is particularly vital for thin sections segments intended to withstand high pressure weights [2].

The compacted graphite cast iron has intermediate mechanical properties between gray cast iron (graphite in a flake form) and spheroidal cast iron where graphite in a nodular form [10]. The microstructures of CGI that like worm shape make CGI have the advantages of gray and ductile cast iron [11]. Thermal conductivity and damping capacity properties are similar to gray iron and tensile strengths are similar to spheroidal cast iron [12-14].

Metallurgical and micromechanical aspects control the resulting microstructure, unsoundness, strength and ductility [15-18]. In this sense, the microstructure parameters are of high order of importance in order to determine the resulting mechanical behavior and other properties of material casting or heat treated of a number of distinctive alloys and materials [15-20].

The first use of CGI was in brake discs for high speed trains, but the essentially usage of CGI diesel engine blocks [21].

Expanding requests for motors with enhanced fuel effectiveness bring down outflows and lighter weight has guaranteed the future utilization of CGI for car motor pieces [2]. One would likewise hope to see additionally innovative work into regions where CGI castings can be actually and cost viably used.

General cooling curve was shown in Fig. 1, when the austenite begins to shape, lowering in temperature between the austenite precipitation and eutectic change occurs and this is because of the austenite dendrites which experienced each other and started to coarsen. At the point where it is sufficient undercooling for the carbon particles to be removed from the liquid; the eutectic response begins. By including inoculant the essential undercooling is diminished. This is trailed by recalescence, where the carbon is devoured by the graphite and both graphite and austenite develop [22]. This is trailed by a lowering in temperature and afterward the strong state change starts. Again the capture in the cooling bend is expected to developed warmth when ferrite and pearlite shapes, some undercooling is required and after that there is some recalescence [23].

2. Experimental investigation

2.1. Material

Compacted graphite cast iron samples were melted at Helwan Company for casting. Four different samples were produced by using medium frequency induction furnace operated at 500-5000 Hz frequency current. The base metal was spheroidal pig iron (GGG70) with the following chemical composition given in Table 1.

<table>
<thead>
<tr>
<th>Chemical composition of pig iron GGG70.</th>
<th>Chemical composition %</th>
</tr>
</thead>
<tbody>
<tr>
<td>Sn</td>
<td>Cu</td>
</tr>
<tr>
<td>GGG 70</td>
<td>0.06</td>
</tr>
</tbody>
</table>

2.2. Test specimen preparation

The four different thickness blocks were cut from the solid block of compacted graphite that shown in Fig. 2, grinded, polished and machined to the required dimensions for different experiments to be investigated. All tests were made for both as cast and heat treated conditions to compare mechanical properties and microstructure before and after heat treatment.

2.3. Microstructure observation

Microstructures of step blocks were examined using optical microscopy. The samples were prepared following standard metallographic procedures. The samples were always cut from the different thicknesses (5, 10, 15 and 20 mm with error ranges ±0.15 mm) of the step block so 16 samples were obtained and mounted in bakalite. After that, samples were grinded manually and polished with diamond suspension with 1 μm particle size; then etched in 2% Nital. During this investigation, ferrite and pearlite percentage were determined by using optical metallography and an image analysis program to ensure that the samples are in the range of compacted cast iron and to obtain the optimum mechanical properties that needed for diesel engine block.

2.4. Tensile strength and hardness test

Tensile test was carried out according to ASTM E 8M-A using universal testing machine LFM-L 20KN at room temperature
25±2°C. Tensile test was carried out for three samples for each condition and the average was taken. Hardness test was carried out using Brinell hardness; three tests were carried out on each specimen to evaluate the Brinell Hardness Number (BHN).

Table 2 shows the chemical composition of CGI; the Mg content varies depending on the pouring time and it is defined as the time between finishing the melting and starting the pouring in the mold.

Compacted graphite cast iron can be subjected to heat treatment to achieve matrix microstructures and required mechanical properties which cannot be obtained in the as-cast condition. The as-cast matrix microstructures usually consist of ferrite or pearlite or combinations of both of them, depending on cast section size and chemical composition of alloy [24].

2.5. Heat treatment of compacted graphite iron

2.5.1. Annealing

The specimen was heated to a temperature of 930 ± 5°C and holding for 2 h; then the furnace was switched off and the samples were taken out after reaching room temperature. The austenitizing temperature was determined using the general cooling curve for CGI [22].

The objective of keeping the specimen at 930 ± 5°C for 2 h is to convert microstructure to austenite and homogenize the structure to eliminate the dendritic structure.

3. Results & discussions

3.1. Pearlite to ferrite contents of CGI

Pearlite to ferrite matrix contents in microstructure of CGI is one of the most important properties, so that the copper (Cu) and tin (Sn) were used as a pearlite stabilizer element. Engine cylinder blocks that contain high carbon equivalent may have approximately 10% pearlite microstructure due to fast cooling rate. Without addition of pearlite stabilizer elements such as copper and tin; the tendency of ferrite matrix microstructure is too high. Since the formation of ferrite matrix is more common in compacted graphite iron than ductile and gray cast iron, more pearlite stabilizer elements such as copper and tin (Cu and Sn) increase the proportion of the pearlite since these elements decreases the carbon diffusion coefficient; and as a result carbon cannot attain a graphite region and is expected to react with iron [25].

Around 1.0% copper and 0.045% Sn were added to achieve about 80% pearlite matrix. During casting of diesel engine blocks, Cu and Sn were added to achieve the desired pearlite content in the delivered compacted graphite iron diesel engine block. Cu and Sn content depend on the geometry of the item which is closely related to the cooling rate [26,27].

Procedure for determining pearlite % for compacted graphite cast iron is as follows:

- Resize the microscopic image.

<table>
<thead>
<tr>
<th>Table 2 – Chemical composition of CGI.</th>
<th>Chemical composition %</th>
<th>Pouring time (min)</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td>C</td>
<td>Si</td>
</tr>
<tr>
<td>Sample 1</td>
<td>3.65</td>
<td>2.55</td>
</tr>
<tr>
<td>Sample 2</td>
<td>3.71</td>
<td>2.53</td>
</tr>
<tr>
<td>Sample 3</td>
<td>3.67</td>
<td>2.50</td>
</tr>
<tr>
<td>Sample 4</td>
<td>3.65</td>
<td>2.51</td>
</tr>
</tbody>
</table>
3.2. Effect of section thickness on nodularity percentage

The amount of spheroidal graphite cast iron depends on the wall thickness of the casting and the Mg content in the casting. Fig. 3 shows the effect of holding time on the residual Mg content.

Mg content in the casting has ranged from 0.045 to 0.021% and as a result different amounts of spheroidal graphite were obtained in the casting, the longer the holding time the lower the residual Mg percentage.

Procedure for deciding nodularity percentage for CGI is as follows:

- Take the image by microscope before etching for the analysis of nodularity and nodule counts.
- Determine graphite area.
- Nodularity % = (average nodule count)/(average graphite area). Take average for three reading for nodule count and graphite area for the same sample.

Holding time displays a reasonable effect on the progression of residual magnesium that identifies with the fading process, Fig. 3. Mg is lost because of its high vapor pressure at this temperature and response with the little measure of oxygen exhibit inside the chamber amid the re-dissolving.
Fig. 6 – Microstructure for different thicknesses after etching (A) 10 mm thick. With pearlite % 63.60 for sample 1, (B) 15 mm thick. With pearlite % 62.17 for sample 1, (C) 20 mm thick. With pearlite % 81.81 for sample 1, (D) 5 mm thick. With pearlite % 67.79 for sample 2, (E) 5 mm thick. With pearlite % 84.40 for sample 3, (F) 15 mm thick. With pearlite % 87.46 for sample 3, (G) 5 mm thick. With.
procedure and holding time at $1450 \pm 10^\circ C$. During the inversion from nodular cast iron to compacted graphite cast iron, happening among the re-melting procedure, Mg residual demonstrates a third order polynomial relation with time in minutes \[28\], as appeared in Eq. (3).

\[
\text{Residual Mg}\% = 0.045 + 0.0007t - 0.0003t^2 + 1E - 05t^3
\] (3)

where, $t$ time in minutes, for $0 < t < 20 \pm 0.15$ mm.

According to cooling rate, faster cooling rate leads to increase in nodularity percentage like that in thicknesses $5 \pm 0.15$ mm that have nodularity % (32.65, 18.70, 20.56 and 23.1) respectively from sample number 1 to sample number 4, this thin thicknesses have higher nodularity %, and in case of lower cooling rate, a decrease in nodularity % was observed in $20 \pm 0.15$ mm thickness that have nodularity % (14.21, 10.76, 11.73 and 13.65) for samples from 1 to 4 respectively.

3.2.1. Effect of residual magnesium on nodularity percentage

The residual magnesium will decrease as the holding time proceeds. In this research, a second order polynomial relation could be derived, correlating nodularity according to ISO 16112 \[29\] standard as a function of the residual Mg at the beginning of the solidification as shown in Fig. 4.

\[
\text{Nodularity } \% = 142.62 - 10364\text{Mg} + 207557 \text{mg}^2
\] (4)

For $0.045 < \text{Mg} < 0.021$, where Mg is in wt. %

3.3. Microstructural results

Graphite morphology significantly affects the mechanical behavior of cast irons with similar matrix compositions. In CGI, the graphite lamella has a blunter shape, causing fewer local stress concentrations, and the lesser graphite continuity gives a longer crack propagation path within the matrix. Fig. 5 reveal the microstructure of different sections of CGI taken from step block after etching to know the content of pearlite to ferrite matrix.

![Graph showing Brinell hardness vs. different wall thicknesses.](image)

**Fig. 7** - Brinell hardness vs. different wall thicknesses.
Fig. 8 – Microstructure for different wall thicknesses after heat treatment and etching (A) 10 mm thick. With ferrite % 32.67 for sample 1, (B) 15 mm thick. With ferrite % 29.66 for sample 1, (C) 20 mm thick. With ferrite % 37.46 for sample 1, (D) 5 mm thick. With ferrite % 63.37 for sample 2, (E) 5 mm thick. With pearlite % 84.40 for sample 3, (F) 20 mm thick. With ferrite % 66.95 for sample 3, (G) 5 mm thick. With ferrite % 17.27 for sample 4 and (H) 20 mm thick. With ferrite % 75.0 for sample 4.
It can be noticed from Fig. 6 that thickness 5 mm in sample number 1 has higher nodularity % about 32.65, this large nodularity due to short pouring time for this sample that about 10.53 ± 0.25 min as well as the fast cooling rate. The microstructure shown in Fig. 6 revealed that the contents of pearlite for thicknesses 10 and 20 mm are above 80% for all samples. The pearlite percentage depends on the pouring time and thickness, longer pouring time results in larger pearlite percentage such as sample number 4 at thickness 20 mm have higher pearlite percentage that equal 90.11% and pouring time 17.53 ± 0.25 min, however lower thicknesses shows smaller pearlite percentage due to the short pouring time that equal 10.53 ± 0.25 min and may be due to carbides precipitation. Lower thicknesses or short pouring time results in dendritic structure within the pearlitic matrix due to faster cooling rate.

3.4. Mechanical properties

The results of hardness and tensile tests are listed in Table 3 and Fig. 7. In particular, Table 3 reports average values of hardness and tensile properties. The analysis of hardness data in Fig. 7 highlights a relationship between hardness and thickness of the castings, for CGI.

3.5. Heat treatment of compacted graphite iron

Compacted graphite iron is usually used as pearlitic matrix for the diesel engine block and ferritic matrix for manifold exhaust. The chemical composition, section thickness and heat treatment are the main factors controlling the microstructure and consequently the mechanical properties.

3.5.1. Effect of heat treatment on pearlite to ferrite contents for compacted graphite iron

The aim of heat treatment is to fades away the defects that appear in microstructure as cast such as carbides and dendrites shapes (i.e. to make microstructure homogeneous) and to obtain ferrite matrix so the heat treatment made in our study was ferritizing annealing, the following microstructure in Fig. 8 showed that the ferrite area fraction is greater than pearlite due to the annealing heat treatment performed.

3.5.2. Effect of heat treatment on mechanical properties

The effect of annealing heat treatment on the mechanical properties (tensile strength and hardness) of the samples as-cast and after heat treatment is shown in Table 3. The maximum tensile strength of the as-cast sample was 716 ± 5 MPa and maximum hardness value of 413 ± 3 BHN with elongation 3.33 ± 0.5%.

Comparing the mechanical properties of annealed heat treated samples with the as-cast sample, the annealed sample showed lower tensile strength 371 ± 5 MPa and lower hardness 165 ± 3 BHN with increase in elongation up to 9.79 ± 0.5%, as shown in Fig. 9. The decrease in tensile strength and hardness are due to the formation of ferrite matrix in the microstructure of the annealed heat treated samples.

4. Conclusions

- Increasing the pouring time from 10 min to 17 min results in lowering the Mg content from 0.031% to 0.021% and lowers the nodule count by about 50% which affect the mechanical properties.
- The pearlite percentage depends on the pouring time and thickness, longer pouring time results in larger pearlite percentage such as sample number 4 with thickness 20 mm have higher pearlite percentage that equal 90.11% and pouring time 17.53 ± 0.25 min, however lower thicknesses shows smaller pearlite percentage due to the short pouring time that equal 10.53 ± 0.25 min and may be due to carbides precipitation. Lower thicknesses or short pouring time results in dendritic structure within the pearlitic matrix due to faster cooling rate in both cases.
- Comparing the mechanical properties of annealed heat treated samples with the as-cast samples, the annealed samples showed lower tensile strength, (about 60–70% of the as-cast samples) and lower hardness, (about 55–60% of the as-cast) with larger elongation %. The decrease in tensile strength and hardness are due to the formation of ferrite matrix in the microstructure of the annealed heat treated samples which soften the structure.
- The nodularity percentage decreased as wall thickness increases for all samples.
- According to pouring time, if lower pouring time such as 10.53 ± 0.25 min that leads to increase in nodularity % like that in thicknesses 5 mm that have nodularity % (32.65, 18.70, 20.56 and 23.1) respectively from sample number 1 to sample number 4, this thin thicknesses have higher nodularity %, and in case of higher pouring time such as 17.53 ± 0.25 min that leads to decrease in nodularity % such that in thicknesses 20 ± 0.15 mm that have nodularity % (14.21, 10.76, 11.37 and 13.65) for sample number 1 to sample number 4 respectively.

Conflicts of interest

The authors declare no conflicts of interest.
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